Report

Revised Impoundments 1 and 2 Treatability Study Results

American Cyanamid Superfund Site

Prepared for

Pfizer Inc. on behalf of Wyeth Holdings LLC

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Executive Summary

This Laboratory Treatability Study has been completed as part of the Focused Feasibility Study (FFS) process for Impoundments 1 and 2 of the American Cyanamid Superfund Site in Bridgewater Township, New Jersey (Site), as defined in the *Revised Focused Feasibility Study Work Plan* (CH2M HILL, 2012e). The *Laboratory Treatability Studies Work Plan for Impoundments 1 and 2, American Cyanamid Superfund Site* (CH2M HILL, 2012d) identifies the following technologies as potentially able to achieve the proposed Remedial Action Objectives (RAOs), described in the *Revised Focused Feasibility Study Work Plan* (CH2M HILL, 2012e)and *Technology Evaluation Work Plan* (CH2M HILL, 2012a):

- Thermal treatment
- Mixing and homogenization with pH adjustment
- Solidification/stabilization

Technologies proposed for treatment of the impoundment materials were evaluated through laboratory treatability studies described in this report. The overall goal of these studies is to evaluate the effectiveness of homogenization, pH adjustment, de-emulsification, solidification/stabilization, and thermal treatment independently and then combined to determine if the subject technologies can achieve the anticipated RAOs for Impoundments 1 and 2 at the Site.

Samples for the treatability study were collected from Impoundment 2 in January 2012 and from Impoundment 1 in February 2012. Samples were collected using a pontoon barge equipped with a vibracore outfitted with a four-inch (outer diameter) stainless steel core barrel and lined with a disposable 6-millimeter-thick polyethylene sleeve. Samples were collected by advancing the vibracore core barrel into the impoundment materials. The cores collected were logged and photographed to document their contents then the different impoundment materials (viscous-rubbery [VR], hard-crumbly [HC], sand- and silt-like, clay-like, and coal aggregate) were segregated into separate containers for shipment to the treatability laboratories for thermal, homogenization, pH adjustment, and solidification /stabilization testing.

The thermal treatment study was premised on two basic objectives:

1) determine the efficacy of thermal treatment for impoundment materials

2) identify and characterize both off-gas and liquid-phase condensate that must be managed if thermal treatment was implemented for treating impoundment contents. The study objectives were successfully accomplished as documented in the extensive observations and data results provided by this report and supporting appendixes.

The thermal study indicated that heating was successful in significantly reducing volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) in the impoundment materials studied. Benzene experienced the most significant decrease in concentration with greater than 90 percent reduction in the VR material treated at 100^{o}C. In addition, the concentration of VOCs in the vapor phase in equilibrium with the thermally treated HC and VR materials were significantly reduced compared to the concentration of VOCs in the vapor phase in equilibrium with the untreated materials. Highly acidic off-gas caused significant corrosion of the carbon steel thermal treatment vessels, aluminum lining, and thermocouples used during the thermal treatment.

Treatment of the material for extended periods of time did not completely dry out the sample. Some physical properties testing were complicated and affected by the high concentrations of VOCs and the matrix of the material before and after heating. Compressive strength testing of the thermally treated HC indicated up to 80 psi was achieved; however, thermal treatment of the VR material was only able to achieve a compressive strength of 9 psi. Even after thermal treatment, VR material that is reheated exhibits liquid behavior as temperatures reach approximately 65°C. The HC material does not show signs of liquefaction up to temperatures of 100°C. When heated to temperatures up to 100^{o}C, layered HC and VR materials do not mix. The VR material showed significant signs of expansion and density reduction during heating, while the HC material showed moderate expansion.

Thermal treatment operations will generate steam and volatilize contaminants contained within the impoundment materials. Data from the impoundment characterization sampling completed in 2010 indicate that the main VOC detected in the impoundment materials include:

- Benzene
- Toluene
- Naphthalene
- Chlorobenzene
- Methyl acetate
- Xylenes (Total)
- Acetone
- Cyclohexane
- Chloromethane
- 1,3-Dichlorobenzene
- Carbon disulfide
- 1,2-Diclorobenzene
- Isopropylbenzene
- Methylcyclohexane
- 1,3,5-Trimethylbenaene
- 1,4-Dichlorobenzene
- Ethylbenzene

In addition, sampling during the treatability study indicates the following compounds are also expected to volatilize during thermal treatment of the impoundment materials:

- 1,2,4-Trimethylbenzene
- Hexane
- Cyclohexane
- Fluorene
- 2-Methylnaphthalene
- Hydrogen sulfide
- Carbonyl sulfide
- Ethyl mercaptan
- Methyl mercaptan
- Dimethyl sulfide
- Ethyl methyl sulfide
- Thiophene
- Isobutyl mercaptan
- 3-Mthylthiophene
- 2-Ethylthiophene
- Sulfur dioxide
- Sulfuric acid
- Acetaldehyde
- Benzylaldehyde
- Butylaldehyde
- Formaldehyde
- Isovaleraldehyde
- Propionaldehyde
- Valeraldehyde

In addition to the compounds above, hydrocarbons of various length and complexity are also expected to volatilize from the impoundment materials during heating.

The mixing, pH adjustment, and solidification/stabilization treatability studies were performed to evaluate different mixing methodologies and mix ratios of several different alkaline pH adjustment materials and pozzolan recipes for solidification/stabilization of both thermally treated and raw impoundment materials. The solidification/stabilization study confirmed that the materials from Impoundment 1 and 2 at the Site could be successfully homogenized, pH adjusted, and solidified using pozzolans.

The basic objectives of this portion of the treatability study were:

1) Evaluate impoundment materials for homogenization
2) Determine the pH of the impoundment materials could be raised to at least 5 to 6 SU and ideally to 11 SU.
3) Evaluate if the impoundment materials could be stabilized/solidified using pozzolan mixtures.

The study confirmed that both raw impoundment 1 and 2 materials could be homogenized; however, while the water layer added to the raw Impoundment 1 material became incorporated with the material during homogenization, the water layer on the raw Impoundment 2 material did not become incorporated with the material during homogenization. Homogenized samples of materials from the Impoundments 1 and 2 were successfully pH adjusted to 10 SU or higher by an addition of Carmeuse Hydrated Lime or high calcium lime kiln dust (HiCal LKD). The hydrated lime did not provide strength to the raw impoundment 2 materials when mixed but the HiCal LKD provided significant strength gains when mixed into the raw impoundment materials. Addition of various blends of pozzolans to the homogenized and pH adjusted impoundment material resulted in strength gains ranging from 59.0 psi to greater than 62.5 psi after only seven days of curing in place.

Bench-scale testing completed to simulate excavation of solidified/stabilized materials from the impoundments indicated that an additional ex-situ remixing step may be required because the strength gains by initial in-situ solidification/stabilization of the impoundment materials are lost after excavation. Remixing was completed using a slurry of LaFarge Portland Cement and water and increased the strength of the remixed impoundment materials to as much as 114 psi after 56 days of curing. The permeability of the solidified/stabilized impoundment materials tested ranged from 1.63×10^{-3} cm/sec to 1.19×10^{-5} cm/sec.

The pozzolan testing of thermally treated Impoundment 1 and 2 materials indicated that the concentration of benzene in the atmosphere above the mixing vessel decreased by approximately two orders of magnitude when compared to benzene concentration above the mixing vessel for the raw impoundment materials. The highest concentrations of VOCs were in the atmosphere above the mixing vessel during the remixing of raw Impoundment 2 materials with up to 44,000,000 $\mu g/m^3$ of benzene.

TCLP and SPLP data were collected for the remixed solidified and stabilized Impoundment materials and benzene was the only compound detected above the TCLP regulatory limit for both the raw and thermally treated materials. The solidified/stabilized raw materials leached approximately one order of magnitude more benzene than the thermally treated impoundment materials. The extent of benzene leaching from the Impoundment materials with the TCLP and SPLP methods was similar.

The results of the laboratory treatability study have not excluded either thermal treatment, mixing, and homogenization with pH adjustment, or a combination of the two technologies as potentially being able to meet the RAOs for the Site. While the ISS studies have indicated that the Impoundment materials can be solidified and stabilized to reduce permeability and provide enough strength for off-site disposal, additional testing may be required to evaluate further reductions in permeability, leaching and volatile emissions. A Tier IV study was completed to evaluate additional pozzolans to reduce permeability and powdered activated carbon (PAC) to reduce leaching and VOC emissions. The pozzolan mixtures tested during the Tier IV studies include the following:

Material Type	Mixture	pH Buffering	De-emulsification	Initial Solidification	Remix / Stabilization
Raw	IMP 2 RM MIX A	6% Hydrated Lime/ 15% water	-	33% SFE / 15% LN / 15% Omni FBC	10% LPC / 8% water
	IMP 2 RM MIX B	6% Hydrated Lime/ 15% water	-	33% SFE / 30% LN	10% LPC / 8% water
Thermally Treated	IMP 2 TT MIX A	-	30% HiCal LKD / 30% water	15% H_2O / 5% LPC / 5% Omni FBC	10% LPC / 8% water
	IMP 2 TT MIX B	-	30% HiCal LKD / 30% water	15% H_2O / 10% LN	10% LPC / 8% water

Notes:
FBC = fluidized bed combustion ash
LN = LaFarge NewCem
LPC = LaFarge portland cement
SFE = spent fullers earth (screened)

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Acronyms and Abbreviations

°C	degree Celsius
µg/kg	microgram per kilogram
µg/L	microgram per liter
µg/m^3	microgram per cubic meter
ACO	Administrative Consent Order
ASTM	American Society for Testing and Materials
BTEX	benzene, toluene, ethyl benzene, and total xylenes
BTX	benzene, toluene, and total xylenes
CA	coal aggregate
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CKD	Cement Kiln Dust
CL	clay-like
cm/s	centimeter per second
COPC	constituent of potential concern
cP	centipoise
FFS	Focused Feasibility Study
FID	flame ionization detector
Focus	Focus Environmental, Inc.
FS	Feasibility Study
H_2S	hydrogen sulfide
HC	hard-crumbly
IDW	investigation-derived waste
IMP	Impoundment
kg	kilogram
LKD	Lime Kiln Dust
LOS	light oily sludge
LSS	Low Solids Stabilization
mg/kg	milligram per kilogram
mg/L	milligram per liter
NAPL	non-aqueous phase liquid
ND	not detected
NR	not reported
NJDEP	New Jersey Department of Environmental Protection
NR	not reported
NS	not sampled

NT	not tested
OBG	O'Brien & Gere
PAH	polyaromatic nuclear hydrocarbon
pcf	pound per cubic foot
Pfizer	Pfizer Inc.
PID	photoionization detector
PPE	personal protective equipment
ppm	part per million
psi	pound per square inch
QAPP	Quality Assurance Project Plan
QC	quality control
QMG	Quantum Management Group
RAO	Remedial Action Objective
RCRA	Resource Conservation and Recovery Act
RECON	Remedial Construction Services, L.P.
RM	raw material
Site	American Cyanamid Superfund Site in Bridgewater Township, New Jersey
SO_2	sulfur dioxide
SOP	standard operating procedure
SPLP	Synthetic Precipitation Leaching Procedure
SSL	sand and silt-like
SU	standard units
SVOC	semivolatile organic compound
T_0	time zero
TCLP	Toxicity Characteristic Leaching Procedure
TIC	tentatively identified compound
T_f	time final
TSF	ton per square foot
TT	thermal treatment
UCS	unconfined compressive strength
USEPA	United States Environmental Protection Agency
VE	Volatile Expansion
VOC	volatile organic compound
VR	viscous-rubbery
WH	Wyeth Holdings LLC
yd^3	cubic yard

SECTION 1

Introduction

This report has been prepared as part of the Focused Feasibility Study (FFS) process for Impoundments 1 and 2 of the American Cyanamid Superfund Site in Bridgewater Township, New Jersey (Site), as defined in the *Revised Focused Feasibility Study Work Plan* (CH2M HILL, 2012e). Impoundments 1 and 2 are on the southern portion of the Site, near the Raritan River (Figure 1-1). This report provides the results of the treatability studies documented in the *Laboratory Treatability Studies Work Plan* (CH2M HILL, 2012d), which presented the technologies potentially able to achieve the proposed Remedial Action Objectives (RAOs), described in the *Revised Focused Feasibility Study Work Plan* and *Technology Evaluation Work Plan* (CH2M HILL, 2012a) as follows:

> The overall goal of these studies was to evaluate the effectiveness of in-situ thermal treatment, homogenization, pH adjustment, and solidification/stabilization, both independently and combined to determine if the subject technologies can achieve the anticipated RAOs for Impoundments 1 and 2. While additional evaluation and studies, such as material compatibility assessments will be required throughout the design and remedial processes, the information provided within this document provides the basis for the next phases of the project as part of the FFS.

The report is organized as follows:

- **Section 1, Introduction:** Describes the Site and regulatory history of Impoundments 1 and 2, and provides a physical description of Impoundments 1 and 2 and characteristics of materials they contain.
- **Section 2, Impoundment Material Collection:** Summarizes the scope of the impoundment material collection activities performed at the Site in 2012, including descriptions of the sample collection and analytical methods, air monitoring, data verification procedures, and variations from the work plan.
- **Section 3, Treatability Testing Overview:** Presents an overview of the laboratory testing program components developed to support completion of the FFS for Impoundments 1 and 2 and details the materials to be used in the proposed studies.
- **Section 4, Thermal Treatment Results:** Discusses the results of the thermal treatment studies conducted.
- **Section 5, Mixing, pH Adjustment, De-emulsification and Solidification and Stabilization Results:** Discusses the results of the mixing, pH adjustment, de-emulsification and solidification and stabilization lab studies conducted.
- **Section 6, Conclusions:** Summarizes the major findings and conclusion**s** of the treatability studies.
- **Section 7, References:** Provides the references cited in this report.

1.1 Site Description

The Site is in the southeastern section of Bridgewater Township, Somerset County, in the north-central portion of New Jersey. The Site is bounded by the New Jersey Transit Railroad to the north, the Raritan River to the south and west, and Somerset Tire Service and Interstate Highway 287 to the east. The Site is divided into five main portions: The North, East, West, and South Areas and the Impoundment 8 Facility. The North Area refers to the portion of the Site within the flood control dike; the East Area is east of Middle Brook; the West Area is west of Bufflehead Road; and the South Area is south of the flood control dike and between Middle Brook and Bufflehead Road; (Figure 1-1). Impoundments 1 and 2 are located within the South Area of the Site.

Figure 1-1

1.1.1 Site History

The Site was used for nearly nine decades to manufacture a range of products including rubber-based chemicals, dyes, pigments, chemical intermediates, petroleum-based products, and pharmaceuticals. A total of 27 impoundments were historically used to store and treat liquid and semi-solid streams generated from the onsite processes, as well as river water for firefighting purposes. Sixteen of the impoundments are regulated pursuant to CERCLA, six impoundments according to RCRA, and five impoundments were used to manage only river water, storm-water or settled silt from this water. Because of past practices, impacts to soil and groundwater have been documented within several areas of the Site. The majority of these impacts occurred within the footprint or in the immediate vicinity of several of the impoundments.

The Calco Chemical Company, which started operations in 1915 manufacturing chemical intermediates and dyes, originally owned the Site. In 1929, American Cyanamid Company purchased the Site and began producing pharmaceuticals and chemicals in the 1930s. During the following decades, the American Cyanamid facility expanded to meet the nation's demands during and immediately after World War II. As production increased from the 1930s through the 1970s, expansion of buildings and support services, as well as raw material and waste management activities were conducted.

In the late 1970s and early 1980s, the facility experienced a significant downsizing. Organics and dye production at the Site was phased out by the early 1980s. The manufacturing of bulk pharmaceuticals continued until all operations at the facility ceased by 1999. In November 1994, the American Cyanamid Company was acquired by the American Home Products Corporation, which changed its name to Wyeth in 2002. In October 2009, Pfizer Inc. (Pfizer) acquired Wyeth. The Site is owned by Wyeth Holdings LLC (WH), which is now a subsidiary of Pfizer. Specific details of the history of Impoundments 1 and 2 at the Site are provided in Section 1.2.

1.1.2 Regulatory Framework

Environmental remediation and restoration activities have been ongoing at the Site under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process. During the development and evaluation of the remedial alternatives for the Site-wide Feasibility Study (FS) (OBG, 1997), it became apparent that handling of the Impoundments 1 and 2 materials was unique and complex. The location of these impoundments within the Raritan River floodplain, along with the acidity and the complex nature of the material, pose significant difficulties in evaluating the technical feasibility of remedial technologies, their implementability, and their ability to meet regulatory requirements.

After review and interaction with stakeholders, including the United States Environmental Protection Agency (USEPA) and the New Jersey Department of Environmental Protection (NJDEP), it was decided by mid-2009 that additional data specific to the Impoundments 1 and 2 materials were needed to complete the evaluations. Therefore, Wyeth and the stakeholders mutually agreed to move Impoundments 1 and 2 into a separate FFS, while moving forward with the Site-wide FS for the remainder of the Site (USEPA, 2008; CH2M HILL2012e).

The USEPA is the lead CERCLA regulatory agency for this Site, with NJDEP providing support, as necessary. Remedial activities at the Site are governed by CERCLA, the Resource Conservation and Recovery Act (RCRA) and the amended Administrative Consent Order (ACO), and the intent of New Jersey's *Technical Requirements for Site Remediation* (NJDEP, 2011). The FFS report for the impoundments will be developed to satisfy the requirements of the 1988 ACO and its subsequent amendment in 1994.

1.2 Impoundments 1 and 2 Features

Impoundments 1 and 2 are in the South Area of the Site (Figure 1-2) and were used to store acid tar residuals. Both Impoundments 1 and 2 are located approximately 700 feet north of the Raritan River. Interstate Highway 287 is approximately 850 feet east of Impoundment 1, and Somerset Tire Service is approximately 400 feet north of the impoundments. The nearest residential areas are approximately 2,000 feet north-east and 1,800 feet east of Impoundment 1.

Figure 1-2

1.2.1 Impoundment 1

Impoundment 1 was constructed in 1956 and was used until 1965 to store waste from a coal light-oil refining process (OBG, 2010). The acidic solid and semi-solid waste materials (pH generally less than 2 standard units [SU]) separated into three distinct layers: a light oily sludge (LOS) layer on the top, a viscous-rubbery (VR) tar layer in the middle, and a hard-crumbly (HC) tar layer at the bottom. About 3 million gallons of the uppermost LOS layer was removed from Impoundment 1 in 1966 and 1967 for offsite energy recovery. In the 1980s, coal aggregate (CA) was deposited into Impoundment 1 to facilitate the excavation of acid tar material for an offsite fuel-blending program. This program was unsuccessful, and coal deposits remain in Impoundment 1. In 1991, a synthetic cover was placed on Impoundment 1. A water cap is maintained over Impoundment 1 for odor control.

Impoundment 1 has a surface area of approximately 2.1 acres and contains:

- An upper layer of VR tar (approximately 900 cubic yards [yd^3])
- A lower layer of HC tar (approximately 13,700 yd^3)
- Additional materials mixed into the VR and HC layers include:
  - Clay-like (CL) material (approximately 2,700 yd^3)
  - Sand and silt-like (SSL) material (approximately 1,900 yd^3)
  - CA (5,000 yd^3) (Focus, 2011)

The total volume of waste in Impoundment 1 is approximately 24,200 yd^3. The volume of the water cap on top of the waste is approximately 9,300 yd^3. Impoundment 1 is characterized by a low pH (average of 3.3 SU) and high concentrations of volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs), including benzene, toluene, xylenes (BTX), naphthalene, and a high degree of residual acidity (Focus, 2011).

1.2.2 Impoundment 2

Impoundment 2 is approximately 1.7 acres in size, was constructed in 1947, and was used until 1956 to store waste from the same refining process (OBG, 2010). Like Impoundment 1, the waste materials discharged to Impoundment 2 also separated into three distinct layers of HC tar, VR tar, and LOS. In 1986 and 1987, about 3 million gallons of LOS material was removed from Impoundment 2 for offsite energy recovery, leaving behind two acid tar layers. Based on the most recent characterization, Impoundment 2 contains:

- An upper layer of VR tar (approximately 10,900 yd^3)
- A lower layer of HC tar (approximately 12,900 yd^3)
- A mixed VR and HC layer (approximately 6,500 yd^3) which is observed between the two distinct layers (Focus, 2011)

The total waste volume of Impoundment 2 is 30,300 yd^3, and foreign materials such as CA or CL materials appear absent in Impoundment 2. A synthetic cover was place on Impoundment 2 in 2012. A water cap is maintained over the synthetic cover for odor control. The volume of the water cap on top of the waste is approximately 10,200 yd^3. Like Impoundment 1, Impoundment 2 is characterized by a low pH (average of 1.5 SU) and high concentrations of VOCs and SVOCs (including BTX, and naphthalene), as well as residual acidity (Focus, 2011).

1.2.3 Physical and Chemical Nature of Acid Tar

As described, Impoundment 2 has two primary layers (VR and HC) with some mixture between the two, and Impoundment 1 has more variability. In addition to the HC and VR layers in both impoundments, Impoundment 1 also contains CA added in the 1980s, CL materials, and SSL materials. The location and depths of these material types vary throughout the impoundment. Descriptions for each impoundment materials identified during the 2010 investigation are as follows (OBG, 2010):

- **VR material:** This material is black and tar-like and characterized by its tackiness and lack of matrix. This material also exhibits an oily sheen; in some instances, the surface appeared glass-like. Generally, this material has a low pH (near 1 SU) and contains high concentrations of VOCs and SVOCs. This material is difficult to handle because of its tackiness; its consistency varies with temperature.

- **HC material:** The HC material is black, often with an oily sheen, and resembles bits of broken asphalt. The HC material generally is highly acidic and contains high concentrations of VOCs and SVOCs. However, in general, the VOC concentrations tend to be an order of magnitude lower, and the SVOC concentrations tend to be 25 percent higher than the concentrations detected in the VR material (OBG, 2010). It is not cohesive and can be broken into small pieces by hand.
- **CA material:** The CA material is grey and fine-grained. While this material is reported to have been placed on the surface of Impoundment 1 in the 1980s, it has shifted deeper into the impoundment.
- **CL material:** CL material is generally grey, similar to the CA, cohesive, and fine-grained. It is generally located in the upper portion of the Impoundment 1 material.
- **SSL material:** This material is a brown, fine-grained, non-cohesive material that is in the middle to lower portion of Impoundment 1.

SECTION 2

Impoundment Material Collection

The following section describes the overall approach used to complete the Impoundments 1 and 2 material collection activities and includes a discussion of the scopes of work, a brief description of the relevant sampling activities, and any significant deviations from the work plan. The standard operating procedures (SOPs) and analytical methods used for the Impoundment material collection are described in more detailed in the *Material Collection Work Plan for Impoundments 1 and 2, American Cyanamid Superfund Site* (CH2M HILL. 2012b).

The following objectives for tar collection were established in support of the treatability testing program:

1) Collect representative material from each impoundment to support testing of individual material types or laboratory derived material mixtures. To support this objective, impoundment materials were field-segregated following collection based on physical characteristics (VR, HC, and so forth). Segregated material of like characteristics collected in each impoundment were then be combined for treatability testing.
2) Collect material from each impoundment that represents the current field conditions. At designated locations in each impoundment, material recovered was immediately containerized following collection.
3) Visually characterize the composition and stratification of tar materials present in each impoundment.

2.1 Sample Collection

A pontoon barge equipped with a vibracore outfitted with a four-inch (outer diameter) stainless steel core barrel and lined with a disposable 6-millimeter-thick polyethylene sleeve was utilized to collect materials from each impoundment. The vessel was assembled onsite and lifted onto the Impoundments by a crane. The vessel was positioned at each of the sampling locations using a Global Positioning System. Due to adverse weather conditions during the first round of sampling in January 2012, the impoundment material collection activities were completed in two separate mobilizations. Subsections 2.1.2 and 2.1.3 describe the sampling collection activities specific to both of these mobilizations and deviations from the work plan. Impoundment 1 and 2 sampling locations are shown on Figure 2-1.

In general, the vibracore barrel was advanced into the impoundment material at each location approximately 1 foot above the clay impoundment liner or where refusal was encountered. The boring logs from the April 2010 impoundment characterization were used to identify the location of the clay liner at each of the collection points. The penetration depth (measured from the top of the material surface [first occurrence beneath the water cap]), water depth, and actual coordinate locations were recorded for each sample location. Additional runs were collected to meet the sample volume requirement for each material type in locations where the material targeted was encountered.

2.1.1 Core Processing

The ends of each section of core were capped and the cores were transported to shore for processing. The processing area was situated at the top of the berms surrounding the impoundments, between Impoundment 1 and 2. The capped sections of core containing the impoundment material were cut open along the length of the core to expose the contents. The first core of each location was screened using a photoionization detector (PID), photographed, and logged to document the extent of different classes of materials present. Additional runs were also photographed and screened for PID detections. A photo log is provided in Appendix B of this report. The different classes of material encountered during processing activities were as follows:

Impoundment 1

- VR Tar
- HC Tar
- CA
- CL Material
- SSL Material

Figure 2-1

Impoundment 2

- VR Tar
- HC Tar

Sample handling procedures were designed to minimize loss of VOCs during processing. Following core characterization, the different materials contained in each core were segregated into separate Teflon-lined, five-gallon polyethylene buckets. Any residual material from the cores that was not placed into the five-gallon buckets was placed back into Impoundment 2. A total of 5 gallons of mixed Impoundment 2 materials were returned to Impoundment 2 during the January 2012 sampling event and 5-gallons of mixed Impoundment 1 materials were returned to Impoundment 2 during the February 2012 sampling event in accordance with the approved Work Plan. Material from both impoundments was placed into Impoundment 2 since a liner had not yet been installed in this impoundment until one month later (March 2012). A composite sample of each class of material was collected from the various sampling locations. The composite samples were then processed for shipment to the corresponding laboratories for treatability testing. Chain-of-custody forms were completed for the samples collected during the impoundment material collection activities. A copy of each chain-of-custody form accompanied the samples during shipment to each of the laboratories. The chain-of-custody form identified each sample container and the analytical parameters for each and listed the field personnel who collected the samples, the project name and number, the name of the analytical laboratory that received the samples, and the method of sample shipment. Samples were shipped via courier during both field events.

2.1.2 Impoundment 1

Due to adverse weather conditions encountered during sampling of Impoundment 2 in January 2012, the material collection at Impoundment 1 was conducted in February 2012. Figure 2-1 depicts the sampling locations for Impoundment 1. Impoundment 1 material was collected at four of the six proposed sampling locations based on barge accessibility to each location and material recovery. Sufficient material was collected from these four locations. The locations sampled were designated IMP1-A, IMP1-B, IMP1-E, and IMP1-L, with material characterization at each location as follows:

- **VR Tar:** IMP1-E only
- **HC Tar:** All fours sampling locations (location IMP1-L exhibited the highest content of HC)
- **Coal Aggregates:** IMP1-A only
- **Clay Like Material:** IMP1-A only
- **Sand/Silt Like Material:** IMP1-B only

Multiple runs were required at each sampling station to achieve the required sample volumes needed for the testing. Material characterization, sampling depths, and number of runs are provided in the sediment cores logs located in Appendix A of this report.

2.1.3 Impoundment 2

Material collection at Impoundment 2 was conducted in January 2012. Figure 2-1 depicts the sampling locations for Impoundment 2. Icy conditions encountered during the sampling event at Impoundment 2 restricted barge accessibility to the six proposed locations. Therefore, three of the proposed six sampling locations were completed during material collection of Impoundment 2. The three sampling points were also re-located based on barge accessibility to each location and material recovery. The locations sampled were designated as IMP2-B, IMP2-J, and IMP2-L, with material characterization at each location as follows:

- **VR Tar:** All three sampling locations
- **HC Tar:** All three sampling locations

Multiple vibracore sampling runs were required to achieve the required sample volumes needed for the testing. Material characterization, sampling depths, and number of runs are provided in the sediment cores logs located in Appendix A of this report.

2.2 Air Monitoring

Air monitoring was conducted during impoundment material collection activities as outlined in the site *Health and Safety Plan (HASP)* (CH2M HILL, 2011) and the *Laboratory Treatability Studies Work Plan* (CH2M HILL, 2012d). Air monitoring was conducted on the vessel and at the core processing area with both a PID and a hydrogen sulfide meter. In addition, Draeger-tube sampling was conducted at the core processing area, upwind and downwind of the work area during material collection, and on the barge during sample collection. Benzene, toluene, ethyl benzene, and total xylenes (BTEX), phenol, chlorobenzene, sulfur dioxide, and carbon disulfide were measured using Draeger tubes each day of sampling. The Draeger-tube monitoring was conducted at times when fugitive emissions were expected to be highest (during core processing). The Draeger-tube downwind air samples were collected from two locations, the first downwind of the impoundments on the top of the berm and the second further downwind (approximately 100 feet) of the impoundments. Upwind samples were also collected both on the barge and at an established distance from the processing area. The results from the air monitoring activities are summarized in Table 2-1. A contingency plan was also established for the collection of Summa canisters at each location, if the results of the Draeger tubes or other hand-held air monitoring equipment showed elevated levels of compounds. Details of the thresholds for triggering Summa canister sampling was provided in the *Laboratory Treatability Studies Work Plan* (CH2M HILL, 2012d). Based on the results of the Draeger tube and other air monitoring, the Summa canister sampling was not required.

2.3 Waste Handling

During collection of materials from Impoundment 2, seven drums of combined tar-impacted personal protective equipment (PPE) and core liners were generated and stored in sealed 55-gallon drums. During collection of Impoundment 1 materials, two drums of PPE and four drums of core liners were generated and stored in sealed 55-gallon drums. All wastes were stored at the Impoundment 8 drum storage area and were properly treated and disposed off-Site. In addition, the following materials were placed back into Impoundment 2 as agreed upon with the USEPA during their Site visit on January 9, 2012.

- Five gallons of porewater and residuals from Impoundment 2 core processing
- Three gallons HC Impoundment 1 material from core processing
- Eleven gallons of porewater and residuals collected during Impoundment 1 core processing

2.4 Quality Control Procedures

SOPs, laboratory methods, and quality control (QC) procedures for the laboratory treatability studies were performed in accordance with the Quality Assurance Project Plan (QAPP) (CH2M HILL, 2012c). Overall, the precision and accuracy of the data, as measured by field and laboratory QC indicators, indicates that the data are usable for project objectives, as qualified.

Table 2-1

SECTION 3

Treatability Testing Overview

This section presents the overall rationale, objectives, and general description of the treatability studies conducted. Previous studies have shown that thermal treatment processes and solidification/stabilization processes are generally applicable for treating a wide array of materials, including coal tars. However, to implement a thermal treatment or solidification/stabilization remedial technology for materials contained in the impoundments, studies regarding the effects of the technologies on the impoundment materials physical and chemical characteristics were required. Therefore, two interrelated bench-scale testing programs were performed to explore thermal treatment, solidification/stabilization and a combination of these technologies. The relationship among proposed thermal treatment and solidification/stabilization technologies is presented on Figure 3-1.

3.1 Thermal Treatment

While several studies have been performed to evaluate the characteristics of the impoundment materials during thermal treatment, prior efforts have focused on combustion-based processes where acid tar served as a fuel source. The thermal treatment completed as part of this treatability test focused on heating the impoundment contents to alter chemical and physical properties but not to the extent where combustion would be supported or induced. The testing program focused on the effects of heat on both physical and chemical characteristics of impoundment materials. The testing program was developed to provide a preliminary assessment of the technical feasibility of heat treatment of impoundment materials.

3.1.1 Rationale and Objectives

As illustrated in the decision flow chart (see Figure 3-1), preliminary laboratory testing of impoundment materials was intended to resolve basic scientific questions regarding the effect of heat on the chemical and physical characteristics of the HC and VR materials within the impoundments. Specific objectives of chemical and physical testing are described as follows.

1) Determine the effect of heating on the chemical composition of impoundment materials. Specific test objectives for VR and HC included:
    a. Evaluate the change in chemical composition of impoundment materials before and after the heating.
    b. Characterize the chemical composition of vapor produced during thermal treatment.
    c. Assess leaching characteristics of impoundment materials following thermal treatment.
    d. Characterize the chemical composition of liquid effluent and condensate resulting from thermal treatment.
2) Determine the effect of heating on the physical properties of impoundment material. Specific test objectives included:
    a. Measure the corresponding change in VR and HC compressive strength before and after thermal treatment.
    b. Determine relative change in HC porosity resulting from thermal treatment.
    c. Measure the effect of temperature on the specific gravity and viscosity of the VR material.
    d. Qualitatively assess apparent change in material characteristics before and after heating.
3) In addition to evaluating the chemical and physical changes that occur during thermal treatment of impoundment materials, the studies also included:
    a. Assess the physical stability of VR material with respect to HC material during heating, and evaluate the potential for homogenization during and after thermal treatment.

b. Prepare sufficient volumes of thermally treated material for additional treatment during the solidification/stabilization study.

3.1.2 Treatability Test Description

The initial thermal treatment study incorporated several types of physical and chemical evaluations of Impoundment 2 materials, as outlined on Figure 3-1. In general, three basic tests (outlined as follows) were completed during the initial thermal study. Detailed discussion of each test is provided in following sections of this report.

1) VR and HC materials were heated in separate temperature-controlled reaction cells. The physical and chemical composition of the impoundment materials, both pre- and post-treatment, were evaluated to assess physical changes, contaminant removal, and off-gas composition.

2) Physical stability assessment of the VR and HC materials as a function of increasing temperature was measured.

3) Viscosity and density of the VR and HC materials was determined as a function of increasing temperature.

3.2 Mixing, pH Adjustment, De-emulsification and Solidification/Stabilization

The mixing, pH adjustment, de-emulsification and solidification/stabilization treatability studies were performed to evaluate different mixing methodologies of both thermally treated and raw impoundment materials. Except in special applications, solidification/stabilization does not destroy the contaminants; rather, it immobilizes (e.g. metals) or volatizes (e.g. volatiles due to heat generation) them. The technology has been used extensively to reduce the leachability of metals and the mobility of organic compounds in soils and tarry materials.

The laboratory studies evaluated mixing methods and several different alkaline materials followed by six different pozzolan recipes for solidification/stabilization. Evaluating these sequenced treatment steps on both the raw and thermally treated impoundment material was proposed due to the significant physical and chemical changes that occurred within the impoundment materials following thermal treatment.

Mixing and homogenization of the impoundment material was performed to create a more uniform material for application of subsequent treatment technologies. The low pH of the impoundment materials required adjustment to a higher pH to allow the pozzolans to properly react with the impoundment materials to enhance the physical and chemical properties. After mixing and pH adjustment, solidification/stabilization treatments were applied to reduce the mobility of constituents in the impoundment materials and increase their strength so that the material can be handled and disposed accordingly. The use of a de-emulsification step for the thermally pre-treated material was necessary to soften/liquefy hardened acid tar material fractions to enhance effective blending operations of added reagents during solidification/stabilization.

The solidification part of the process may involve the addition of binders to adsorb liquids and increase strength. The stabilization part of the process involves adding chemicals to react with the impoundment material to form less soluble and less mobile compounds.

Solidification/stabilization treatment typically accomplishes:

- Reduced contaminant solubility by formation of sorbed species or insoluble precipitates
- Improved physical characteristics of the waste (i.e., compressive strength, permeability, lower leachability)
- Decreased exposed surface area across which mass transfer loss of contaminants may occur
- Lessened contact between transport fluids and contaminants by reducing the permeability of the material

Figure 3-1

3.2.1 Rationale and Objectives

Specific objectives of mixing, pH adjustment, de-emulsification and solidification/stabilization treatability studies are described as follows.

1) Mixing and homogenization and pH adjustment of raw and thermally treated impoundment materials:
    a. Determine the ability of mixing and homogenization to develop a homogeneous blend of VR and HC impoundment materials.
    b. Evaluate blending agents to adjust the pH of VR and HC materials with a target of 5 to 6 SU.
    c. Evaluate de-emulsification agents with thermally pretreated Impoundment material to soften and liquefy the material for homogenization prior to pozzolan reagent addition.
    d. Evaluate temperature rise from exothermic chemical reactions between the impoundment materials and alkaline additives along with emissions produced during the mixing and homogenization and pH adjustment of impoundment materials.
    e. Assess operational conditions for potential field implementation.
2) Solidification/stabilization of raw and thermally treated impoundment materials:
    a. Evaluate mixing various solidification/stabilization reagent materials to achieve the proposed RAOs.
    b. Evaluate temperature rise from exothermic chemical reactions between the waste and pozzolan additives along with emissions produced during solidification/stabilization reagent mixing.
    c. Stabilize and solidify impoundment materials.
    d. Evaluate changes in the chemical properties of the impoundment materials after solidification/stabilization.
    e. Evaluate the changes in physical properties of the impoundment materials after solidification/stabilization, such as unconfined compressive strength (UCS) and permeability.

3.2.2 Treatability Test Description

The mixing, pH adjustment, de-emulsification and solidification/stabilization portion of the study was completed in three stages. These stages include the following:

1) Mixing, homogenization, and pH adjustment/de-emulsification of raw and thermally treated Impoundments 1 and 2 materials.
2) Pozzolan screening of homogenized and pH adjusted raw and thermally treated Impoundments 1 and 2 materials.
3) Optimization of promising pozzolans identified during the screening stage of the study to evaluate the final physical and chemical properties of the solidified and stabilized materials.

SECTION 4

Thermal Treatment Results

Thermal treatment of impoundment materials for the removal of volatile and malodorous compounds offers substantial advantage as part of the remedial strategy. Heat removes the volatile and malodorous compounds, and depletion of these compounds from the impoundment materials results in significant beneficial physical changes in the characteristics of the impoundment materials.

4.1 Baseline Material Chemical Characterization

Prior to conducting any studies on the impoundment materials, batches of HC and VR materials were collected from Impoundment 2 in January 2012, as described in Section 2 of this report. Thermal testing involved evaluation of the physical and chemical response of Impoundment 2 HC and VR materials. As described in Section 3, this study was completed using Impoundment 2 materials only. Since there is no appreciable difference in chemical or physical attributes of the HC and VR materials between the impoundments, observations gained during this study will be representative of heating effects on materials common to both Impoundment 1 and 2. The HC and VR materials were homogenized in separate large tubs by kneading the individual materials until each material was well mixed. Photographs of homogenized VR and HC impoundment materials are included in Appendix B. Samples of the homogenized VR and HC impoundment materials were collected and analyzed in triplicate for VOCs, SVOCs, and metals. In addition to chemical characterization of the untreated impoundment materials, the air above the impoundment materials was evaluated for VOCs, malodorous compounds, aldehydes, and inorganic acid gases. Sample collection and analytical methods used are located in the *QAPP for the Laboratory Treatability Studies of Impoundment 1 and 2 Materials* (CH2M HILL 2012c).

4.1.1 Viscous-rubbery Material – VOCs

Selected VOCs detected in Impoundment 2 VR material are summarized in Table 4-1; results are presented in order of decreasing mass fraction. In general, results are consistent with previous analytical testing of VR material and confirm that VR is primarily composed of aromatic and substituted aromatic hydrocarbons, including BTEX. Among these compounds, ethyl benzene was present in the lowest concentration. Results also indicated that naphthalene (a polyaromatic hydrocarbon [PAH]) and 1,2-dichlorobenzene are each present at appreciable concentrations in the VR material. Detailed tables of results are located in the *Results for Treatability Evaluation of Site Materials the American Cyanamid Superfund Site in Bridgewater Township, New Jersey prepared* (TerraTherm 2012), located in Appendix C.

TABLE 4-1
Laboratory Treatability Studies Report
Selected Volatile Organics Detected in Untreated Impoundment 2 VR Material

Compound	Minimum Concentration (micrograms per kilogram [µg/kg])	Maximum Concentration (µg/kg)	Average Concentration (µg/kg)
Benzene	18,100,000	26,300,000	21,100,000
Toluene	5,570,000	7,510,000	6,306,667
Naphthalene	3,060,000	3,810,000	3,410,000
Total Xylene[1]	1,590,000	2,270,000	1,886,667
1,2-Dichlorobenzene	1,650,000	2,120,000	1,860,000
1,2,4-Trimethylbenzene	301,000	610,000	533,667
sec-Butylbenzene	504,000	575,000	479,000
Cumene	353,000	550,000	458,667
1,3,5-Trimethylbenzene	301,000	466,000	390,000
Ethylbenzene	107,000	110,000	108,500

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.

4.1.2 Viscous-rubbery Material – SVOCs

Presented in order of decreasing mass fraction, SVOCs detected in homogenized Impoundment 2 VR material are summarized in Table 4-2. Analytical results are consistent with previous characterization efforts and confirm that the composition of VR material within Impoundment 2 is predominantly made up of VOCs, as previously summarized. All SVOCs observed are components commonly found in coal-derived tar. Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm, located in Appendix C.

TABLE 4-2
Laboratory Treatability Studies Report
Selected Semi-Volatile Organics Detected in Pretreated Impoundment 2 VR Material

Compound	Minimum Concentration (µg/kg)	Maximum Concentration (µg/kg)	Average Concentration (µg/kg)
Fluorene	941,000	2,390,000	1,547,000
2-Methylnaphthalene	243,000	589,000	384,000
Acenaphthene	39,100	94,800	61,500
Aniline	22,300	58,700	40,500

4.1.3 Viscous-rubbery Material – Total Metals

The metals results of the initial chemical characterization of the Impoundment 2 VR material are summarized in Table 4-3. Results of total metals analysis for VR material were consistent with historical observations reported during previous investigations (Focus, 2011). Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm, located in Appendix C.

TABLE 4-3
Laboratory Treatability Studies Report
Selected Metals Detected in Untreated Impoundment 2 VR Material

	Untreated VR Concentration (mg/kg)		
Compound	**Minimum**	**Maximum**	**Average**
Metals			
Aluminum	319	360	334
Arsenic	ND	ND	ND
Barium	7.51	8.16	7.87
Chromium	2.76	5.23	4.25
Copper	23.5	28.6	25.3
Iron	881	1,120	998
Lead	62.4	67.7	65
Nickel	5.12	5.42	5.27
Mercury	1.71	1.96	1.81

4.1.4 Viscous-rubbery Material – Equilibrium Vapor Head Space

Vapor in equilibrium with VR material under ambient conditions was also evaluated for VOCs, acid gases, aldehydes, and malodorous compounds. Analytical results of headspace sampling are summarized in Table 4-4. Similar to the solid phase, VOC analysis results include BTX as the dominant organic compounds that evolve from the VR material when exposed to ambient air. Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm, located in Appendix C.

TABLE 4-4
Laboratory Treatability Studies Report
Selected Compounds Detected in Untreated Impoundment 2 VR Material Headspace Samples

VOCs	Ambient Time Zero (T_0) (micrograms per cubic meter [µg/m^3])	Aldehydes	Ambient Time Zero (T_0) (parts per million [ppm])
Carbon Disulfide	333,000	Hydrobromic Acid	< 0.5
Benzene	110,000,000	Hydrochloric Acid	< 1
Toluene	14,000,000	Hydrofluoric Acid	< 2
m,p-Xylene	1,000,000	Nitric Acid	< 0.6
Total Xylene[1]	1,000,000	Phosphoric Acid	< 2
		Sulfuric Acid[2]	< 2
Reduced Sulfur Compounds	**Ambient Time Zero (T_0) (µg/m^3)**	**Inorganic Acids**	**Ambient Time Zero (T_0) (ppm)**
Hydrogen Sulfide	640	Acetaldehyde	0.6
Carbonyl Sulfide	200	Benzaldehyde	< 0.02
Methyl Mercaptan	< 20	Butyraldehyde	0.07
Ethyl Mercaptan	< 25	Crotonaldehyde	< 0.03
Dimethyl Sulfide	680	Formaldehyde	< 0.07
Carbon Disulfide[3]	48,000	Isovaleraldehyde	2.7
Isopropyl Mercaptan	< 31	Propionaldehyde	0.1
tert-Butyl Mercaptan	< 37	Valeraldehyde	< 0.02
n-Propyl Mercaptan	< 31		
Ethyl Methyl Sulfide	170		
Thiophene	66		
Isobutyl Mercaptan	< 37		
Diethyl Sulfide	< 37		
n-Butyl Mercaptan	< 37		
Dimethyl Disulfide	39		
3-Methylthiophene	< 40		

Notes:
[1.] Total xylene calculated by the sum of ortho, meta, and para –xylene concentration detected.
[2.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.
[3] Carbon disulfide detected by more than one analytical method; highest concentration detected is presented.

4.1.5 Hard-crumbly Materials – VOCs

Results of VOC analysis for Impoundment 2 HC material are summarized in Table 4-5. Like the previous data summary, HC results are presented in order of decreasing mass fraction. Overall VOC results are consistent with previous analytical testing and clearly illustrate that the HC material is predominantly composed of BTX and naphthalene. The HC material does differ slightly from the VR in VOC composition, most notably in the lower concentration of 1,2-dichlorobenzene. The mass distribution and detection of substituted aromatic hydrocarbons, including cumene, 1,2,4-trimethyl benzene and 1,3,5-trimethyl benzene, in the HC also differs from the VR concentrations reported. Unlike the VR, ethyl benzene and sec-butylbenzene were not detected in the HC samples analyzed. Benzene is the predominant compound detected in the HC; however, the observed concentration was significantly higher than measured in VR samples. On average, benzene in HC measured 33,100,000 µg/kg, versus 21,100,000 µg/kg in the VR. Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm, located in Appendix C.

TABLE 4-5
Laboratory Treatability Studies Report
Selected Volatile Organics Detected in Untreated Impoundment 2 HC Material

Compound	Minimum Concentration (µg/kg)	Maximum Concentration (µg/kg)	Average Concentration (µg/kg)
Benzene	21,400,000	42,400,000	33,100,000
Toluene	4,260,000	9,120,000	6,866,667
Total Xylene[1]	1,670,000	2,530,000	2,203,333
Naphthalene	1,490,000	2,070,000	1,816,667
Cumene	769,000	778,000	773,500
1,2,4-Trimethylbenzene	557,000	610,000	741,000
1,2-Dichlorobenzene	551,000	662,000	606,500
1,3,5-Trimethylbenzene	607,000	625,000	616,000

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.

4.1.6 Hard-crumbly Materials – SVOCs

Presented in order of decreasing mass fraction, SVOCs detected in Impoundment 2 HC material are summarized in Table 4-6. Analytical results are consistent with previous characterization efforts and confirm that the composition of HC material within Impoundment 2 is composed predominantly of VOCs as previously summarized. Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm located in Appendix C.

TABLE 4-6
Laboratory Treatability Studies Report
Selected Semi-Volatile Organics Detected in Untreated Impoundment 2 HC Material

Compound	Minimum Concentration (µg/kg)	Maximum Concentration (µg/kg)	Average Concentration (µg/kg)
Fluorene	133,000	223,000	187,667
2-Methylnaphthalene	39,100	60,600	51,700
Acenaphthene	5,930	11,600	8,617

4.1.7 Hard-crumbly Materials – Total Metals

The metals results of the initial chemical characterization of the Impoundment 2 HC material are summarized in Table 4-7. Results of total metals analysis for HC material are similar to VR sample results with the two exceptions: arsenic was not detected in the VR material, and the concentration of mercury in the HC material is two times the concentration detected in the VR material. Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm, located in Appendix C.

TABLE 4-7
Laboratory Treatability Studies Report
Selected Metals Detected in Untreated Impoundment 2 HC Material

Compound	Untreated HC Concentration (mg/kg)		
	Minimum	Maximum	Average
Metals			
Aluminum	164	286	226
Arsenic	1.41	5.95	3.10
Barium	5.31	6.23	5.74
Chromium	4.16	8.36	6.26
Copper	12.5	15.4	13.6
Iron	529	776	648
Lead	59	76.1	68.8
Nickel	3.57	6.13	4.75
Mercury	2.74	8.68	4.93

4.1.8 Hard-crumbly Materials – Equilibrium Vapor Head Space

Vapor in equilibrium with HC material from Impoundment 2 was also evaluated for VOCs, acid gases, aldehydes, and malodorous compounds under ambient temperature conditions during the treatability study. Analytical results of headspace sampling are summarized in Table 4-8. Like the solid-phase VOC analysis results, BTX are the dominant organic compounds that evolve from the HC when exposed to ambient air. Detailed tables of results are located in the Treatability Study Report prepared by TerraTherm, located in Appendix C.

TABLE 4-8
Laboratory Treatability Studies Report
Selected Compounds Detected in Untreated Impoundment 2 HC Material Headspace Samples

VOCs	Ambient Time Zero (T_0) (µg/m^3)	Aldehydes	Ambient Time Zero (T_0) (ppm)
Carbon Disulfide	550,000	Hydrobromic Acid	< 0.5
Benzene	180,000,000	Hydrochloric Acid	< 1
Hexane	390,000	Hydrofluoric Acid	< 2
Toluene	22,000,000	Nitric Acid	< 0.6
m,p-Xylene	1,400,000	Phosphoric Acid	< 2
Total Xylene[1]	1,400,000	Sulfuric Acid[2]	6.4
Reduced Sulfur Compounds	**Ambient Time Zero (T_0) (µg/m^3)**	**Inorganic Acids**	**Ambient Time Zero (T_0) (ppm]**
Hydrogen Sulfide	64,000	Acetaldehyde	0.2
Carbonyl Sulfide	1,500	Benzaldehyde	< 0.02
Methyl Mercaptan	540	Butyraldehyde	< 0.03
Ethyl Mercaptan	< 510	Crotonaldehyde	< 0.03
Dimethyl Sulfide	13,000	Formaldehyde	< 0.07
Carbon Disulfide[3]	770,000	Isovaleraldehyde	2
Isopropyl Mercaptan	< 620	Propionaldehyde	< 0.04

TABLE 4-8
Laboratory Treatability Studies Report
Selected Compounds Detected in Untreated Impoundment 2 HC Material Headspace Samples

Reduced Sulfur Compounds	Ambient Time Zero (T_0) ($\mu g/m^3$)	Inorganic Acids	Ambient Time Zero (T_0) (ppm]
tert-Butyl Mercaptan	< 740	Valeraldehyde	< 0.02
n-Propyl Mercaptan	< 620		
Ethyl Methyl Sulfide	2,600		
Thiophene	2,800		
Isobutyl Mercaptan	< 740		
Diethyl Sulfide	< 740		
n-Butyl Mercaptan	< 740		
Dimethyl Disulfide	< 390		
3-Methylthiophene	< 800		

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
[2.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.
[3.] Carbon disulfide detected by more than one analytical method; highest concentration detected is presented.

4.2 Thermal Treatment Material Evaluation

The thermal treatment study focused on VR and HC material collected from Impoundment 2 only, as this material was judged to have the most challenging physical and chemical properties of the two impoundments. As defined in the treatability testing work plan, thermal treatment of Impoundment 2 VR and HC material was performed in the laboratory at both 90 degrees Celsius (°C) and 100°C. Study temperatures were selected to evaluate the relative differences in material treatment above and below the boiling point of water. The impoundment materials were heated for approximately 24 days during the 90°C test and 14 days during the 100°C test. In total, four thermal treatment evaluations were performed in support of the treatability study:

- VR material heated at 100°C
- HC material heated at 100°C
- VR material heated at 90°C
- HC material heated at 90°C

A photograph of the experimental setup employed is in Appendix B and detailed in KEMRON work plan. Briefly, each material and temperature condition was tested as follows:

- A known sample mass (VR or HC material) was placed in a sealed carbon steel treatment cell (box reactor). This box reactor was then placed in a temperature controlled oven.
- Sweep gas was supplied to the box reactor headspace and the displaced vapors were conveyed to a condenser equipped with a receiver for condensate collection. Vapor exiting the condenser was routed to the laboratory fume hood.
- The box reactor and the sample were then heated to the target temperature and monitored using both qualitative and quantitative analytical methods.
- When VOC concentration of vapor exiting the box reactor decreased to below 1,000 ppm (measured by laboratory FID), heating operations were terminated; the box reactor was removed from the oven, allowed to cool, and materials were subsequently sampled.

Routine vapor screening (daily) consisted of qualitative measurements of VOCs, hydrogen sulfide, and sulfur dioxide obtained through direct-reading hand-held instruments equipped with FID, PID, and parameter-specific detectors. Periodic vapor samples for VOCs, acid gases, aldehydes, and malodorous compounds were also

collected for analysis by a third-party laboratory. Results of thermal treatment are presented in the following sections. A detailed discussion of the methods and materials used to test Impoundment materials, and the analytical monitoring applied is summarized below and is also found within Appendix C of this report.

4.2.1 Trial Box Reactor

A trial box reactor was set up to evaluate the proposed testing methods. In addition, the trial box reactor provided data regarding how the impoundment material would behave when heated to 100°C. Shortly after heating commenced, the trial reactor experiment was terminated because at approximately 92°C, the VR material inside the box reactor expanded beyond the volume of the box reactor and VR was pushed through the vapor effluent line into the condenser. After ending the test, the box reactor was removed from the oven while it was still near 90°C. The heated VR material was fluid with a consistency similar to molten chocolate. A photograph of the heated VR material from the first trial run is contained in Appendix B. The entire system was broken down and cleaned to remove tar residues from the equipment.

Expansion of the VR upon heating was unforeseen. As a result, a simple evaluation was conducted in the laboratory to quantify the change in VR volume upon heating. A known volume of VR material was added to a graduated cylinder and heated in a laboratory drying oven maintained at 105 °C. During this test, the VR material expanded to three times the initial volume placed in the cylinder. Based on knowledge gained during the first trial box reactor study, a second trial box reactor study was conducted using a smaller initial mass (700 grams) of VR and a larger reactor that could accommodate the expansion of the VR material. The change in both treatment cell and initial sample mass allowed for successful heating of the VR to 100°C in the trial test.

The effects of heating of VR materials in the trial test cell were evident immediately. As the treatment cell temperature increased and approached 70°C, condensate formation within the condenser was observed. With increasing temperature, the volume and rate of condensate accumulation also increased. Condensate produced initially exhibited two distinct phases with clearly separated interfaces, but with increasing treatment time, a third phase became evident (see photograph in Appendix B). Specific gravity of the upper two layers was less than 1 gram per cubic centimeter and both were insoluble in distilled water. Specific gravity of bottom layer was not measured; however, this condensate phase was fully miscible with distilled water. The three-phase condensate was consistent with observations from the bulk heating operations which are described in Section 5.2. Condensate produced in the trial was opaque and ranged in color from light tan to dark brown. Comparatively, condensate produced in the bulk heating was consistently clear and colorless to lightly colored (yellow hue) upon initial generation. With increasing time, the base layer in the bulk treatment condensate became white and opaque, which did not occur in trial treatment cell observations.

Condensate generated in the trial test cell was collected and segregated based on relative specific gravity. Three fractions corresponding to the top, middle, and bottom layers were subsequently submitted for chemical analysis. In general, the types and relative distribution of VOCs and SVOCs observed corroborates measurements performed on the VR material prior to thermal treatment. Selected analytical results, presented in Table 4-9, confirm that the top layer of condensate was composed primarily of BTX, naphthalene, dichlorobenzene, and similar substituted monoaromatic hydrocarbons. A comprehensive listing of condensate sample results including tentatively identified compounds (TICs) is provided in Appendix C.

TABLE 4-9
Laboratory Treatability Studies Report
Summary of Selected Volatile Organics Detected in Trial Reactor Condensate Layers

Compound	Bottom Layer (micrograms per liter [µg/L])	Middle Layer (µg/L)	Top Layer (µg/L)
Benzene	454,000	4,090,000	270,000,000
Toluene	64,100	724,000	58,600,000
Total Xylene[1]	8,300	100,000	8,120,000
Naphthalene	6,170	82,500	6,460,000

TABLE 4-9
Laboratory Treatability Studies Report
Summary of Selected Volatile Organics Detected in Trial Reactor Condensate Layers

Compound	Bottom Layer (micrograms per liter [µg/L])	Middle Layer (µg/L)	Top Layer (µg/L)
1,2-Dichlorobenzene	6,040	62,300	4,410,000
1,2,4-Trimethylbenzene	1,300	13,100	1,170,000
1,3,5-Trimethylbenzene	793	8,940	374,000
Acetone	120,000	102,000	ND
pH[2]	2.6	3.2	2

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
[2.] pH was measured using pH paper and is approximate.

Vapor screening was conducted after the condenser using a handheld FID while test cell temperatures increased and for the entire period of isothermal treatment. Once the cell temperature approached 50°C, several measurements were performed; however, all attempts saturated the FID detector, indicating the vapor from the trial test cell was highly enriched in VOCs. Detector saturation occurred at 50,000 ppm, and as such, VOC monitoring results for the trial were reported as greater than the 50,000 ppm saturation threshold for the instrument. The trial box reactor was ended after 10 days of heating after the VOC concentration measure by the FID was reduced to 758 ppm. After 10 days of heating, measurements confirmed that one third of the initial VR sample mass was removed by heating operations in the trial reactor.

In addition to demonstrating the experimental setup, the trial test was also commissioned to demonstrate the integration of quantitative analytical methods to monitor the composition of vapor produced during the thermal treatment tests. As previously described, the concentration of VOCs, inorganic acids, aldehydes, and malodorous compounds were measured in the untreated VR material (Section 4.1.4 and 4.1.8). Vapor data of the raw impoundment materials at ambient temperature was collected to represent baseline conditions (T_0) of the atmosphere over the VR material. Headspace samples for malodorous compounds were collected at T_0, at the time the impoundment material reached temperature (day 1), and after 2 days of heating. Because the vapor sample collection method employed for T_0 conditions was not feasible for the treatment cell configuration, vapor was collected directly after the condenser to analyze the requisite suite of VOCs, inorganic acids, aldehydes, and malodorous compounds. Vapor samples were collected before and during the trial test according to the following timetable:

- T_0 – to represent the baseline conditions of untreated impoundment material
- Day 1 – to represent initial heating ramp
- Day 2 – to provide an interim assessment of aldehyde, inorganic acid, and malodorous compound removal
- Day 5 – to represent VOCs present following sustained- and steady-state treatment conditions

Selected analytical results are summarized in Table 4-10. A comprehensive listing of vapor sample results, including TICs, is provided in Appendix C. Overall, vapor samples collected to characterize the VR material emission during trial thermal treatment were consistent with results generated during analytical evaluation of the solid-phase material. Vapor under ambient and elevated temperature was predominantly composed of benzene, toluene, and carbon disulfide which were detected as major components of the untreated VR material at ambient temperatures.

Several reduced sulfur compounds that were not observed at ambient conditions were detected in vapor from the trial reactor. Most notable was the presence of hydrogen sulfide, which was detected at 41,000,000 µg/m^3 in the day 2 vapor sample. Aldehyde analysis confirmed the presence of acetaldehyde, benzaldehyde, and butyraldehyde in the ambient T_0 samples; however, with the exception of acetaldehyde (observed in the day 2

sample), aldehyde species were not detected during any of the sampling events associated with VR material heating. Interestingly, formaldehyde, a reagent reportedly used in the historical manufacturing process that produced the impoundment materials, was not detected in the baseline or test cell samples associated with VR heating. Sulfuric acid was the only inorganic acid detected in the head space sample during the trial reactor study. Sulfuric acid was not detected at the detection limit of 1,000 µg/m^3 at T_0 and increased to 3,400 µg/m^3 at day 1 and increased to 4,700 µg/m^3 at day 2. The day 1 and 2 sulfuric acid data may be biased low because the inorganic acid samples are collected using a detector tubes and during collection, and sulfuric acid saturated the sorbent in the detector tube.

TABLE 4-10
Laboratory Treatability Studies Report
Summary of Selected Compounds Detected in Baseline and Trial Reactor Vapor During Heating

Compound	Ambient T_0 Concentration[1] (µg/m^3)	Day 1 Concentration (µg/m^3)	Day 2 Concentration (µg/m^3)	Day 5 Concentration (µg/m^3)
VOCs				
Benzene	2,500,000	16,000,000	NS[2]	1,200,000
Toluene	34,000	200,000	NS	80,000
Chloromethane	54,000	ND[3]	NS	15,000
Acetone	ND	150,000	NS	140,000
Carbon Disulfide	85,000	200,000	NS	6,000
Hexane	14,000	96,000	NS	ND
Cyclohexane	9,000	31,000	NS	ND
Malodorous Compounds				
Hydrogen Sulfide	ND	2,000,000	41,000,000	NS
Carbonyl Sulfide	630	1,700,000	33,000	NS
Methyl Mercaptan	ND	18,000	470,000	NS
Ethyl Mercaptan	ND	ND	48,000	NS
Dimethyl Sulfide	2,400	18,000	21,000	NS
Carbon Disulfide	130,000	1,000,000	51,000	NS
Isopropyl Mercaptan	ND	ND	16,000	NS
Ethyl Methyl Sulfide	330	ND	ND	NS
Thiophene	1,500	ND	ND	NS
Isobutyl Mercaptan	370	ND	ND	NS
3-Methylthiophene	160	ND	ND	NS
2-Ethylthiophene	67	ND	ND	NS
Aldehydes				
Acetaldehyde	1.2	< 0.08	0.6	NS[2]
Benzaldehyde	0.2	< 0.08	< 0.08	NS
Butyraldehyde	0.4	< 0.09	< 0.09	NS
Crotonaldehyde	< 0.09	< 0.09	< 0.09	NS
Formaldehyde	< 0.08	< 0.08	< 0.08	NS
Isovaleraldehyde	< 0.08	< 0.08	< 0.08	NS
Propionaldehyde	0.3	< 0.08	< 0.08	NS
Valeraldehyde	0.2	< 0.09	< 0.09	NS

TABLE 4-10
Laboratory Treatability Studies Report
Summary of Selected Compounds Detected in Baseline and Trial Reactor Vapor During Heating

Compound	Ambient T_0 Concentration[1] ($\mu g/m^3$)	Day 1 Concentration ($\mu g/m^3$)	Day 2 Concentration ($\mu g/m^3$)	Day 5 Concentration ($\mu g/m^3$)
		Inorganic Acids		
Hydrobromic Acid	< 1	< 1	< 1	NS
Hydrochloric Acid	< 1	< 1	< 1	NS
Hydrofluoric Acid	< 1	< 1	< 1	NS
Nitric Acid	< 1	< 1	< 1	NS
Phosphoric Acid	< 1	< 1	< 1	NS
Sulfuric Acid[4]	< 1	3.4	4.7	NS

Notes:
[1.] Sample size is 40 ml vial with approximately 5.0 grams of sample.
[2.] NS = Not sampled
[3.]ND = Not detected
[4.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.

Trial Reactor Conclusions

As outlined in the *Laboratory Treatability Studies Work Plan* (CH2M HILL, 2012d), the purpose of the trial test using VR material was to confirm that experimental and analytical methods proposed for testing were feasible given the physical and chemical characteristics of the impoundment materials. In light of the results summarized in previous sections and detailed in the attached laboratory report (Appendix C), the conclusions from the trial reactor study are as follows:

- With the exception of qualitative vapor measurements using the FID, change in vapor-phase chemical composition resulting from heating could be reliably measured over the study duration.
- When heated, VR material undergoes volume expansion. Heating to 105°C resulted in a volume swell of roughly 3:1 compared to ambient laboratory temperature.
- Vapor generated during heating produced a three-phase liquid condensate; the speciation of VOCs detected in the condensate correlated directly with VOCs detected during solid-phase analysis of untreated VR material.
- Under the test conditions established, vapor-phase samples were successfully collected and analyzed for parameters of interest including VOCs, malodorous compounds, aldehydes, and inorganic acid gas.
- Vapor generated during heating was highly enriched in VOCs; the speciation of vapor phase VOCs correlated well with VOCs detected during solid phase analysis of untreated VR material.
- After 10 days of heating, nearly one third of the initial VR mass sample mass was removed by heating operations in the test cell.

4.2.2 Impoundment Material Testing

Detailed thermal treatability testing of the impoundment materials commenced in early July and progressed in general accordance with the testing protocol outlined in the work plan. Because the VR material expanded to three times its initial volume in the trial box reactor study, the expansive properties of the HC material were evaluated prior to setup of the box reactors for the main study by placing a known volume of HC in a graduated cylinder and heating it above 100°C. Although the HC material did expand slightly, the observed volume change was substantially smaller than change observed during VR material heating. Heating of impoundment materials followed the prescribed ramp with intermediate isothermal holds at 30°C, 50°C, 70°C, and 90°C or 100°C for the

qualitative screening of vapor (for hydrogen sulfide, sulfur dioxide, and VOCs) exiting the condenser assembly. Once the test reactor reached the desired treatment temperature, vapor exiting the condenser was screened daily.

Screening results for VOCs were used to assess the relative extent of treatment with time in each box reactor. To prevent saturation of screening instrument detectors, early term samples were diluted with ambient air to estimate the approximate concentration for each parameter. Dilution factors varied during the course of heating operations to account for the wide range of total VOCs present in the vapor exiting the condenser system. Supplemental discussion of measurement techniques for vapor screening is contained in Appendix C.

The vapor exiting the condenser in each box reactor was also sampled for VOCs, malodorous compounds, aldehydes, and acid gas for laboratory analysis. Based on observations from heating during the trial reactor test and the need to extend the heating duration, the vapor sampling schedule was altered to ensure that samples over the entire duration of testing would be collected. The revised vapor sampling schedule was:

- Sample 1 (T_0) – representing ambient or baseline conditions for untreated impoundment material
- Sample 2 – completion of day 1 of heating to document change during initial temperature ramp
- Sample 3 – when total VOC concentration measured by FID decreased below 50,000 ppm
- Sample 4 – when total VOC concentration measured by FID decreased below 1,000 ppm
- Sample 5 (Time Final [T_f]) – representing the return of treated impoundment material to ambient temperature

A synopsis of treatment observations for each material condition tested follows in subsequent sections.

Hard-crumbly Material – 90°C

Hard crumbly material was successfully heated to the target temperature in the box reactor. Following approximately 4 hours of heating, the HC material within the reactor reached the target temperature of 90°C. Initial production of condensate in the collection vessel was observed when the box reactor achieved approximately 80°C; sample heating produced a two-phase condensate. The top layer was pale yellow, thick, and had an oil-like appearance. The bottom layer resembled the top but was colorless. Generally, the pH of the top layer remained higher (3 to 4 SU) than the bottom layer (2 to 3 SU).

The vapor produced during heating was screened using an FID and hydrogen sulfide and sulfate hand-held meters during temperature ramp-up, and then daily after the impoundment material reached temperature. The results of the vapor screening conducted during the testing of HC material treated at 90°C are provided in Table 4-11. A graph representing the headspace screening data during the treatment of the HC material at 90°C is depicted as Figure 4-1.

TABLE 4-11
Laboratory Treatability Studies Report
Vapor Screening Summary for HC Material Treated at 90°C

Day		Hydrogen Sulfide (H_2S) (ppm)	Sulfur Dioxide (SO_2) (ppm)	VOCs (ppm)
Ambient (Untreated)		32.0	19.8	206,895
Day 0	30°C	30.0	21.3	254,226
	50°C	16.0	> 150	> 500,000
	70°C	483	> 150	413,600
	90°C	308	> 150	197,305
Day 1		321	25.9	240,350
Day 2		485	13.7	268,000
Day 3		422	23.0	10,062

TABLE 4-11
Laboratory Treatability Studies Report
Vapor Screening Summary for HC Material Treated at 90°C

Day	Hydrogen Sulfide (H_2S) (ppm)	Sulfur Dioxide (SO_2) (ppm)	VOCs (ppm)
Day 4	86.0	32.5	8,849
Day 5	114	18.6	15,630
Day 6	202	1.20	79,662
Day 7	254	29.0	30,688
Day 8	184	98.0	20,876
Day 9	202	149	20,403
Day 10	360	142	4,766
Day 11	2.00	11.2	4,246
Day 12	148	138	4,304
Day 13	115	98.7	2,785
Day 14	106	118	3,876
Day 15	98.0	104	6,781
Day 16	88.0	58.6	2,578
Day 17	101	72.0	3,447
Day 18	21.0	64.8	1,127
Day 19	310	124	1,214
Day 20	120	87.0	1,370
Day 21	272	23.1	2,099
Day 22	NR[1]	NR	NR
Day 23	57.8	47.2	1,287
Day 24	35.0	2.2	833

[1] NR = Not Reported.

The work plan assumed seven days would be required to treat the HC impoundment materials to reduce the VOCs; however, 10 days of heating was required during the trial reactor study to treat the VR material at 100°C. The HC material treated at 90°C required 24-days before the VOC concentration in the off-gas was reduced to below 1,000 ppm. The initial mass of HC material used in this test was 1,997.5 grams and heating resulted in the loss of 468 grams of the initial HC sample mass. Overall, heating of the HC material caused a 23.5 percent mass reduction compared to pre-treatment conditions. Once cooled, visual inspection of the treated HC material revealed that the surface was hard (brittle), uneven, and friable; there was also evidence of air pockets between the surface layer and the underlying material. Photographs of the pre- and post-treatment HC material are located in Appendix B. With increasing depth, moist material was encountered in the bottom layers of the sample. Heat-treated HC material possessed a characteristic tar-like odor, and the interior of the box reactor showed significant surface corrosion and pitting.

Figures 4-1

Figure 4-2

Viscous-rubbery Material – 90°C

To accommodate expansion of VR material during heating, large box reactors were employed for testing at 90°C and 100°C. To further prevent the chance for expulsion of the heated VR material from the reactor through the off-gas line during heating, approximately 8 inches of vertical headspace in the reactor was retained following placement of material in the test cell. As previously described, the box reactor temperature was ramped to the 90°C target temperature with monitoring activities performed at temperatures of 30°C, 50°C, 70°C, and 90°C. At a temperature of 57.5°C, condensate was first noticed in the condensate collection vessel. As with the HC material, a two-phase condensate was observed. Liquid in the top layer was a clear, thick, and oily; properties of the bottom layer appeared similar except the layer was opaque and milky white. Generally, the pH of the top layer remained higher (3-4 SU) than the bottom layer (2-3 SU). Following approximately 1.5 days of heating, the VR material within the reactors reached a temperature of 90°C.

The vapor produced during heating was screened using an FID and hydrogen sulfide and sulfate hand-held meters during temperature ramp-up, and then daily after the impoundment material reached temperature. The results of the vapor screening conducted during the testing of VR material treated at 90°C are provided in Table 4-12. A graph representing the headspace screening data during the treatment of the VR material at 90°C is depicted as Figure 4-2.

TABLE 4-12
Laboratory Treatability Studies Report
Vapor Screening Summary for VR Material Treated at 90°C

Day		H_2S (ppm)	SO_2 (ppm)	VOCs (ppm)
Ambient (Untreated)		2.00	0.00	428,970
Day 0	30°C	0.00	1.30	51,460
	50°C	6.7	5.00	123,410
	70°C	2.5	3.00	431,290
	90°C	284	150	500,000
Day 1		620	30.1	384,360
Day 2		330	0.00	261,900
Day 3		370	20.3	28,310
Day 4		434	5.00	31,526
Day 5		320	5.10	43,958
Day 6		435	5.40	39,842
Day 7		428	0.900	13,181
Day 8		385	12.7	12,988
Day 9		274	10.8	12,200
Day 10		18.0	0.200	11,026
Day 11		14.0	0.200	9,978
Day 12		6.00	0.400	8,752
Day 13		122	2.400	13,580
Day 14		2.00	0.200	8,210
Day 15		97.0	5.50	1,843
Day 16		68.0	13.7	2,333
Day 17		73.0	6.7	3,188

TABLE 4-12
Laboratory Treatability Studies Report
Vapor Screening Summary for VR Material Treated at 90°C

Day	H_2S (ppm)	SO_2 (ppm)	VOCs (ppm)
Day 18	17.0	0.700	2,017
Day 19	181	11.0	3,025
Day 20	118	16.6	3,033
Day 21	201	11.7	1,472
Day 22	NR	NR	NR
Day 23	483	2.80	711

[1] NR = Not Reported

The work plan assumed seven days would be required to treat the HC materials to reduce the VOCs; however, the trial reactor test indicated that at least 10 days were required to treat the VR material at 100°C. After 23 days of heating at 90°C, vapor screening results indicated that vapor exiting the condenser assembly had fallen below the 1,000-ppm threshold established for termination of the heating test. Heating resulted in the loss of 969 grams of the initial VR sample mass present. Overall, heating of the VR material represented a 37.9 percent mass reduction compared to pre-treatment conditions. Treated VR material possessed a rubbery texture and was removed from the box reactor as rubbery matt. Photographs of the pre- and post-treatment VR material are located in Appendix B. The interior of the box reactor showed significant corrosion with both rusting and some pitting.

Hard-crumbly Material – 100°C

Testing of the HC material at a target treatment temperature of 100°C was performed in accordance with the testing protocol previously outlined and defined in the work plan. The vapor produced during heating was screened using an FID and hydrogen sulfide and sulfate hand-held meters during temperature ramp-up, and daily after the impoundment material reached temperature. The results of the vapor screening conducted during the testing of HC material treated at 100°C are provided in Table 4-13.

TABLE 4-13
Laboratory Treatability Studies Report
Vapor Screening Summary for HC Material Treated at 100°C

Day		H_2S (ppm)	SO_2 (ppm)	VOCs (ppm)
Ambient (Untreated)		29.0	43.4	154,864
	30°C	10.0	97.4	394,696
	50°C	216	18.0	54,030
Day 0	70°C	191	43.2	284,304
	90°C	191	98.1	283,500
	100°C	460	30.0	162,335
Day 1		169	37.3	230,820
Day 2		109	19.7	18,960
Day 3		95.2	152	27,881
Day 4		105	128	40,490
Day 5		113	165	15,162
Day 6		358	129	12,458
Day 7		21.0	48.0	2,553

TABLE 4-13
Laboratory Treatability Studies Report
Vapor Screening Summary for HC Material Treated at 100°C

Day	H_2S (ppm)	SO_2 (ppm)	VOCs (ppm)
Day 8	102	87.0	7,166
Day 9	100	98.0	5,987
Day 10	109	134.0	6,879
Day 11	44.0	132	7,919
Day 12	34.0	39.0	5,742
Day 13	12.0	4.50	4,680
Day 14	1.0	163	943[1]

Notes:
[1.] Comprehensive analysis of box reactor study data (including post thermal treatment residuals) suggests that Day 14 vapor screening measurement may be biased low.

After 14 days of heating at 100°C, vapor screening results indicated that vapor exiting the condenser assembly had fallen below the 1,000-ppm threshold established for termination of the heating test. However, subsequent evaluation of analytical test results (presented in Section 4.3.3 [Table 4-20]) suggest that the 100°C test was prematurely terminated. Although several FID measurements were made to support test shutdown (vapor less than 1000 ppm) the decrease in total VOC measured in previous days was much smaller compared to the decrease observed between day 13 and 14. Early termination of heating at 100°C had no negative effect on test results or in fulfillment of study objectives. However, since the heating duration was not sufficient to fully exhaust VOCs from the solid matrix, the overall mass reduction demonstrated in the 100°C study is lower than what might be expected if the heating period was extended.

In general, treatment time at 100°C was faster than at 90°C; the mechanism for increased mass removal observed at the higher temperature may be related to steam stripping effects since water present in the sample could theoretically boil. Heating resulted in the loss of 378 grams of the initial HC sample mass present. Overall, heating of the VR material represented a 25 percent mass reduction compared to pre-treatment conditions. Despite a treatment temperature that was 10°C higher, visual inspection of the treated material revealed that the treated HC material was very similar to the treated HC material from the 90°C test. The noted exception was that significantly more water remained in the reactor bottom. Photographs of the post-treatment HC material are located in Appendix B. The interior of the box reactor showed significant surface corrosion and pitting.

Thermal monitoring results confirmed the box reactor temperature was maintained at or slightly above 100°C for the duration of testing. Observation of water in the box reactor was unexpected since treatment temperatures were hot enough and held for a sufficient period to allow complete water removal from the system. Although not confirmed in this phase of testing, HC material appears to form a skin or membrane when heated. The hard and undulating surface of the cooled HC treatment cells suggests that steam or vapor bubbles may have been trapped beneath the skin formed on the HC surface. The membrane was not impermeable since significant condensate volume was produced in both HC reactors tested. Further study of phenomena limiting the removal of water during heating may be required to definitively resolve this testing observation.

Viscous-rubbery Material – 100°C

Isothermal treatment of VR material at 100°C was conducted for approximately 13 days using a large box reactor with sufficient headspace for material expansion during heating. The vapor produced during heating was screened using an FID and hydrogen sulfide and sulfate hand-held meters during temperature ramp-up, and daily after the impoundment material reached temperature. The results of the vapor screening conducted during the VR treated at 100°C test are provided in Table 4-14.

TABLE 4-14
Laboratory Treatability Studies Report
Vapor Screening Summary for VR Material Treated at 100°C

Day		H_2S (ppm)	SO_2 (ppm)	VOCs (ppm)
Ambient (Untreated)		0.00	5.90	60,048
	30°C	0.00	2.90	48,460
	50°C	2.00	11.3	150,000
Day 0	70°C	0.00	11.1	210,400
	90°C	5.00	115	450,000
	100°C	482	28.7	201,585
Day 1		0.400	16.0	164,000
Day 2		316	9.80	22,740
Day 3		244	6.10	15,112
Day 4		356	12.8	49,173
Day 5		306	96.4	2,895
Day 6		290	66.4	26,680
Day 7		32.0	103	9,023
Day 8		198	148	11,985
Day 9		247	120	11,026
Day 10		148	137	11,412
Day 11		108	78.2	5,223
Day 12		14.0	13.8	4,926
Day 13		16.0	11.2	833

Notes:
[1.] Comprehensive analysis of box reactor study data (including post thermal treatment residuals) suggests that Day 14 vapor screening measurement may be biased low.

As noted previously, in light of analytical data received several weeks after completion of testing, the 100°C box reactor studies were terminated prematurely. The observed decrease in FID concentration from day 12 to day 13 was considerably larger than many previous and while not outside the realm of previous measurements, laboratory analytical results of post treatment residuals support heating was incomplete for the VR material tested. Regardless of test duration, a substantial mass of VOCs was removed through heating as evidence in both condensate production and the analytical results performed on residual solids after heating (summarized in Section 4.3.3.2). Overall, thermal treatment resulted in the loss of 732 grams of the initial VR sample mass present, which was a 29 percent mass reduction compared to pre-treatment conditions. At the conclusion of testing (that is, FID less than 1,000 ppm), the heating furnace was turned off and the reactor was allowed to cool. Once cooled, the treated VR material was stiff and could not be easily removed from the test reactor. To aid material removal, the reactor was reheated for approximately one hour at 100°C to make the material more viscous. Treated VR material was then immediately removed from test reactor using a stainless steel spoon. The interior of the box reactor showed significant surface corrosion pitting.

Figures 4-3

Figure 4-4

4.2.3 Thermal Treatment Results

The treatability tests previously described in this section were conducted to explore specific objectives as related to the potential application of thermal processes for the treatment of materials contained within Impoundments 1 and 2. Comprehensive data tables with supporting text narratives appear within supporting reports compiled by TerraTherm and their subcontract laboratory (Appendix C). To streamline presentation of study findings, selected results from the box reactor studies performed on VR and HC materials are grouped and reported by test parameter and analytical method. Where applicable, data summaries were also developed to allow direct comparison of material changes imparted by heating and overall efficacy of the thermal treatment process simulated in the laboratory.

Condensate Characterization

Each layer of condensate collected during treatment was individually analyzed for VOCs and SVOCs. For consistency in data presentation among supporting documents, sample nomenclature assigned by the analytical laboratory is retained in Table 4-15. Specifically, the top layer is identified in the laboratory report as "solid" (it consisted of a NAPL) and the bottom layer as the "water" layer. As noted in condensate samples from the trial reactor, BTX are the predominate compounds identified in the condensate. In addition to BTX, the following compounds were also detected in the water fraction only:

- Acetone
- Aniline
- 1,4-Dichlorobenzene
- *ortho*-Cresol
- *meta*-Cresol
- *para*-Cresol
- Phenol
- Pyridine

These results confirm that the speciation of VOCs detected in the condensate is directly correlated with VOCs detected during solid phase analysis of untreated impoundment materials. A comprehensive listing of condensate sample results including TICs is provided in Appendix C.

TABLE 4-15
Laboratory Treatability Studies Report
Summary of Selected Organic Compounds Detected in Box Reactor Condensate

Compound	VR 90°C		VR 90°C		HC 100°C		VR 100°C	
	NAPL[1] (µg/kg)	Water (µg/L)	NAPL (µg/kg)	Water (µg/L)	NAPL (µg/kg)	Water (µg/L)	NAPL (µg/kg)	Water (µg/L)
Benzene	20,200,000	1,470,000	38,400,000	1,310,000	12,700,000	1,230,000	9,840,000	1,090,000
Toluene	2,990,000	136,000	10,500,000	124,000	3,650,000	180,000	2,530,000	135,000
Total Xylene[2]	ND[3]	ND	3,640,000	9,390	747,000	11,500	703,000	10,400
Naphthalene	ND	ND	6,950,000	10,200	ND	ND	ND	ND
2-Methylnaphthalene	80,300	55.50	234,000	43.40	220,000	50.40	720,000	45.70
Fluorene	40,400	25.90	70,600	9.62	139,000	20.90	453,000	23.70

Notes:
1. NAPL = Non-aqueous phase liquid
2. Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
3. ND = Not detected

Treated Material Chemical Characterization

Analyses for total VOCs, SVOCs, and TAL metals for treated impoundment materials were performed in triplicate upon completion of laboratory heating. A comprehensive data set of detected analytes for each analytical method is presented in Appendix C. Minimum, maximum, and average values for selected analytes in VR and HC samples tested at 90°C and 100°C are presented in Table 4-16, Table 4-17, Table 4-18, and Table 4-19.

TABLE 4-16

Laboratory Treatability Studies Report

Summary of Selected Organic Compounds in Treated VR90 and HC90 Impoundment Materials

Compound	VR 90°C Concentration (µg/kg)			HC 90°C Concentration (µg /kg)		
	Minimum	Maximum	Average	Minimum	Maximum	
Volatile Organic Compounds						
Benzene	184,000	201,000	190,333	282,000	656,000	471,333
Toluene	369,000	381,000	377,000	454,000	938,000	701,000
Naphthalene	4,320,000	4,670,000	4,456,667	1,570,000	1,820,000	1,703,333
Total Xylene[1]	535,000	554,000	544,667	464,000	769,000	633,333
1,2-Dichlorobenzene	1,210,000	1,290,000	1,250,000	413,000	538,000	484,667
1,2,4-Trimethylbenzene	213,000	230,000	222,333	66,000	89,100	79,100
sec-Butylbenzene	102,000	113,000	109,000	51,700	77,500	68,567
Cumene	187,000	192,000	188,667	93,700	135,000	117,233
1,3,5-Trimethylbenzene	77,000	85,000	81,733	50,700	53,100	51,900
Ethylbenzene	9,710	10,100	9,880	38,500	38,500	38,500
Semi-Volatile Organic Compounds						
Flourene	242,000	505,000	391,333	205,000	402,000	281,667
2-Methylnaphthalene	52,700	106,000	83,367	53,200	101,000	77,100
Acenaphthene	3,540	3,610	3,577	3,240	3,620	3,387
Aniline	9,810	10,000	9,913	8,990	10,000	9,380

Notes:

[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.

TABLE 4-17

Laboratory Treatability Studies Report

Summary of Selected Metals in Treated VR90 and HC90 Impoundment Materials

Compound	VR 90°C Concentration (mg /kg)			HC 90°C Concentration (mg /kg)		
	Minimum	Maximum	Average	Minimum	Maximum	Average
Metals						
Aluminum	3,290	4,040	3,710	392	505	467
Arsenic	0.16	0.17	0.167	18.8	43.3	27.7
Barium	7.26	8.5	7.94	6.99	11.2	8.81
Chromium	4.30	50.3	20.2	6,910	14,000	9,517
Copper	24.7	27.0	25.9	92.1	201	129.
Iron	6,660	21,100	11,556	24,300	49,700	34,233
Lead	73.1	74.1	73.5	66.7	90.1	78.1
Nickel	4.94	5.65	5.34	3,420	6,700	4,623
Mercury	0.91	1.13	1.01	1.67	3.14	2.44

TABLE 4-18

Laboratory Treatability Studies Report

Summary of Selected Organic Compounds in Treated VR100 and HC100 Impoundment Materials

Compound	VR 100°C Concentration (µg/kg)			HC 100°C Concentration (µg/kg)		
	Minimum	Maximum	Average	Minimum	Maximum	Average
Volatile Compounds						
Benzene	30,000	31,500	30,600	304,000	479,000	390,000
Toluene	148,000	163,000	154,333	351,000	528,000	443,333
Naphthalene	2,240,000	2,510,000	2,413,333	522,000	730,000	620,000
Total Xylene[1]	248,000	259,000	253,667	235,000	338,000	288,333
1,2-Dichlorobenzene	2,710	3,010	2,853	858	882	871
1,2,4-Trimethylbenzene	133,000	143,000	137,333	31,600	43,300	37,600
sec-Butylbenzene	104,000	114,000	109,000	29,600	36,800	33,400
Cumene	73,800	78,000	75,867	41,600	56,100	49,267
1,3,5-Trimethylbenzene	79,000	84,300	82,000	22,900	29,700	26,567
Ethylbenzene	3,450	3,820	3,627	12,900	18,800	15,967
Semi-Volatile Compounds						
Flourene	221,000	436,000	317,667	76,800	102,000	90,667
2-Methylnaphthalene	52,600	108,000	73,900	19,100	25,000	22,633
Acenaphthene	1,630	1,640	1,637	724	1,620	1,028
Aniline	4,800	11,100	8,163	2,010	2,090	2,050

Notes:

[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.

TABLE 4-19

Laboratory Treatability Studies Report

Summary of Selected Metals in Treated VR100 and HC100 Impoundment Materials

Compound	VR 100°C Concentration (mg/kg)			HC 100°C Concentration (mg/kg)		
	Minimum	Maximum	Average	Minimum	Maximum	Average
Metals						
Aluminum	4,330	5,550	4,757	514	578	544
Arsenic	0.150	0.150	0.150	14.0	26.3	22.1
Barium	7.17	9.94	8.16	10.3	11.4	10.7
Chromium	3.06	9.22	5.82	7,310	7,890	7,640
Copper	42.1	314	164	126	150	135
Iron	2,700	4,110	3,287	23,600	28,000	26,133
Lead	59.2	76.9	65.4	73.1	98.4	86.5
Nickel	4.8	12	7.25	4420	4740	4633
Mercury	0.21	0.33	0.273	2.39	2.77	2.52

Additionally, results of TCLP testing for each material and condition tested are presented in Table 4-20. Results of Synthetic Precipitation Leaching Procedure (SPLP) testing, which was also performed during the treatability, are summarized in Appendix C. The TCLP results indicate that the VR material treated at 100°C and the HC impoundment material treated 90°C exceeded TCLP criteria for chromium. In addition, the VR material treated at 100°C also exceeded TCLP criteria for pyridine and benzene. The exceedance for chromium is attributed to corrosion of the metal components in contact with the impoundment material during treatment. In the HC material chromium concentration increased from an untreated average of 6.3 mg/kg to a treated average of 9,517 mg/kg in samples heated to 90°C and an average of 7,640 mg/kg in samples heated to 100°C. Although the magnitude of total change was significantly lower, chromium concentration also increased in the VR impoundment material. The average chromium concentration for VR preheating was 4.2 mg/kg in the untreated material which increased to an average of 20 mg/kg in the 90°C test and 5.8 mg/kg in the 100°C test.

The VR material treated at 100°C also exceeded TCLP criteria for pyridine and benzene. This may be attributed to the treatment being ended before complete VOC removal. Although the VOC concentration in the vapor decreased to below 1,000 ppm on day 13 of treatment (Table 4-14), the concentration of VOCs in the vapor on day 12 was 4,926 ppm. This is a steeper decline in VOC concentration than was observed in either of the other three treatment tests. Thermal treatment testing on VR and HC materials at 90°C and 100°C reduced the concentration of major VOCs (BTX compounds) an average 91 percent Treatment of VR at 90°C and HC at both 90°C and 100°C also resulted in material that successfully passed TCLP criteria for all organic compounds. Additional treatment time for VR material at 100°C is expected to produce material that achieves TCLP criteria.

TABLE 4-20
Laboratory Treatability Studies Report
TCLP Results – VR and HC Impoundment Materials Post-heating

Compound	TCLP Criteria (milligrams per liter [mg/L])	VR 90°C - TCLP (mg/L)	HC 90°C - TCLP (mg/L)	VR 100°C - TCLP (mg/L)	HC 100°C - TCLP (mg/L)
Metals					
Barium	100	0.13	0.051	0.02	0.055
Cadmium	1.0	ND[1]	0.0028	ND	0.028
Chromium	5.0	0.068	186[2]	ND	233
Lead	5.0	0.028	0.42	ND	0.46
Selenium	1.0	ND	0.034	ND	ND
Silver	5.0	0.0033	ND	0.0032	0.0085
Mercury	0.2	ND	0.00009	ND	0.0004
SVOCs					
1,4-Dichlorobenzene	7.5	ND	ND	0.083	0.062
Cresols	200	ND	ND	0.241	0.391
Pyridine	5.0	ND	2.37	ND	6.47
m,p-Cresol	200	ND	ND	0.214	0.359
o-Cresol	200	ND	ND	0.026	0.033
VOCs					
2-Butanone	200	ND	ND	0.0094	0.301
Benzene	0.5	0.08	2.52	0.564	13

Notes:
1. ND = Not Detected
2. Shaded cells exceeded respective TCLP threshold concentration.

Vapor Sampling

As previously described, vapor samples for VOCs, malodorous compounds, aldehydes, and acid gases were collected following the condenser five times during sample heating. Regardless of material type tested, vapor generated during heating was highly enriched in VOCs; the speciation of vapor phase VOCs correlated well with VOCs detected during solid-phase analysis of untreated VR material. With increasing time at each treatment temperature, VOCs present in the vapor exiting the condenser decreased significantly as anticipated.

In general, results of the malodorous compound testing revealed that the analytes were observed at the greatest concentration within days 1 through 3 of treatment. A significant concentration decrease with increasing time was observed for all parameters. The highest concentrations were seen as hydrogen sulfide, carbon disulfide, and thiophenes. Hydrogen sulfide remained in the sample after treatment at elevated concentrations. A comprehensive data set of detected analytes for each analytical method is additionally presented in Appendix C.

Selected results for malodorous compounds and VOCs detected are presented for VR and HC material at 90°C and 100°C in Table 4-21, Table 4-22, and Table 4-23. Results of aldehyde and acid gas testing for box reactor vapor effluent were consistent with analytes observed during completion of the trial reactor previously described. These compounds were not major constituents in the vapors from the impoundment materials during and after treatment and results for aldehyde, and acid gas samples are presented in Appendix C.

TABLE 4-21
Laboratory Treatability Studies Report
Summary of Selected Vapor Phase Malodorous Compounds Detected During Thermal Treatment of Impoundment Materials at 90°C

Material	Compound	Ambient T_0 (µg/m³)	DAY 1 (µg/m³)	< 50,000 ppm (µg/m³)	< 1,000 ppm (µg/m³)	Ambient T_f (µg/m³)
VR 90°C	Hydrogen Sulfide	640	1,400,000	2,700,000	1,500,000	2,900
	Carbonyl Sulfide	200	2,900	7,200	13,000	3,100
	Methyl Mercaptan	ND	28,000	41,000	16,000	100
	Ethyl Mercaptan	ND	2,700	7,800	2,700	47
	Dimethyl Sulfide	680	26,000	10,000	2,500	510
	Carbon Disulfide	48,000	1,100,000	100,000	22,000	3,300
	Ethyl Methyl Sulfide	170	1,800	ND	520	120
	Thiophene	66	3,900	6,000	2,200	360
HC 90°C	Hydrogen Sulfide	64,000	32,000,000	420,000	110,000	28,000
	Carbonyl Sulfide	1,500	26,000	12,000	7,300	310
	Methyl Mercaptan	540	700,000	7,300	980	150
	Ethyl Mercaptan	ND	55,000	980	140	ND
	Dimethyl Sulfide	13,000	84,000	20,000	9,000	2,400
	Carbon Disulfide	770,000	620,000	25,000	12,000	1,300
	Ethyl Methyl Sulfide	2,600	5,500	3,000	980	350
	Thiophene	2,800	19,000	43,000	8,300	3,900

TABLE 4-22
Laboratory Treatability Studies Report
Summary of Selected Vapor Phase Malodorous Compounds Detected During Thermal Treatment of Impoundment Materials at 100°C

Material	Compound	Ambient T_0 (µg/m³)	DAY 1 (µg/m³)	< 50,000 ppm (µg/m³)	< 1,000 ppm (µg/m³)	Ambient T_f (µg/m³)
VR 100°C	Hydrogen Sulfide	640	120,000	6,100,000	3,600,000	470,000
	Carbonyl Sulfide	200	1,100	31,000	95,000	11,000
	Methyl Mercaptan	ND	17,000	47,000	11,000	320
	Ethyl Mercaptan	ND	4,200	10,000	2,700	ND
	Dimethyl Sulfide	680	3,900	19,000	4,000	23,000
	Carbon Disulfide	48,000	48,000	54,000	18,000	6,200
	Ethyl Methyl Sulfide	170	1,300	ND	ND	2,300
	Thiophene	66	9,500	14,000	6,800	23,000
HC 100°C	Hydrogen Sulfide	64,000	1,200,000	4,500,000	350,000	22,000
	Carbonyl Sulfide	1,500	12,000	6,000	10,000	16,000
	Methyl Mercaptan	540	91,000	20,000	2,900	230
	Ethyl Mercaptan	ND	22,000	3,300	ND	74
	Dimethyl Sulfide	13,000	63,000	31,000	24,000	1,400
	Carbon Disulfide	770,000	110,000	15,000	16,000	7,800
	Isopropyl Mercaptan	ND	5,900	ND	ND	99
	tert-Butyl Mercaptan	ND	11,000	ND	ND	240
	n-Propyl Mercaptan	ND	3,100	ND	ND	ND
	Ethyl Methyl Sulfide	2,600	9,400	ND	2,500	340
	Thiophene	2,800	56,000	32,000	30,000	5,900

TABLE 4-23
Laboratory Treatability Studies Report
Summary of Selected Vapor Phase VOCs Detected During Thermal Treatment of Impoundment Materials

Material	Compound	Ambient T_0 (µg/m³)[1]	DAY 1 (µg/m³)	< 50,000 ppm (µg/m³)	< 1,000 ppm (µg/m³)	Ambient T_f (µg/m³)[1]
VR 90°C	Acetone	ND[2]	ND	ND	28,000	ND
	Carbon Disulfide	333,000	610,000	98,000	3000	ND
	Benzene	110,000,000	34,000,000	64,000,000	420,000	920,000
	Toluene	14,000,000	340,000	4,600,000	71,000	650,000
	m,p-Xylene	1,000,000	ND	ND	900	140,000
HC 90°C	Acetone	ND	1,200,000	110,000	27,000	ND
	Carbon Disulfide	550,000	ND	ND	5,300	ND
	Benzene	180,000,000	110,000,000	7,300,000	310,000	2,000,000
	Toluene	22,000,000	17,000,000	4,600,000	150,000	1,600,000
	m,p-Xylene	1,400,000	430,000	620,000	19,000	710,000

TABLE 4-23
Laboratory Treatability Studies Report
Summary of Selected Vapor Phase VOCs Detected During Thermal Treatment of Impoundment Materials

Material	Compound	Ambient T_0 (µg/m³)[1]	DAY 1 (µg/m³)	< 50,000 ppm (µg/m³)	< 1,000 ppm (µg/m³)	Ambient T_f (µg/m³)[1]
VR 100°C	Acetone	ND	ND	140,000	81,000	68,000
	Carbon Disulfide	330,000	160,000	ND	7,300	4,500
	Benzene	110,000,000	12,000,000	9,800,000	400,000	150,000
	Toluene	14,000,000	990,000	3,900,000	510,000	280,000
	m,p-Xylene	1,000,000	44,000	240,000	130,000	97,000
HC 100°C	Acetone	ND	64,000	72,000	150,000	ND
	Carbon Disulfide	550,000	5,300	ND	16,000	ND
	Benzene	180,000,000	1,200,000	6,000,000	3,900,000	ND
	Toluene	22,000,000	810,000	2,500,000	3,000,000	1,400,000
	m,p-Xylene	1,400,000	200,000	330,000	870,000	190,000

Notes:
[1.] Ambient temperature samples (T_0)were evaluated using a different analytical approach than samples measured during active reactor heating operations. Final samples of treated material therefore may not compare unfavorably to vapor samples collected on the final day of box reactor heating.
[2.] ND = Not Detected

4.2.4 Thermal Treatability Test Conclusions

The thermal treatability study confirmed that controlled heating of VR and HC materials at the Site was successful in significantly reducing the VOCs and SVOCs concentrations. A summary of treatment efficacy by material type and temperature is illustrated on Figure 4-1 for the HC material and Figure 4-2 for the VR material. Data plotted for each test condition represent the average pre- and post-treatment concentration for BTX and naphthalene, which were determined to be the predominant compounds detected in the impoundment materials. Overall, thermal treatment resulted in the following removal efficiencies:

- HC material treated at 90°C
  - Benzene was reduced from an average of 33,100,000 to 471,133 µg/kg, or 99.58 percent removal
  - Toluene was reduced from an average of 6,866,667 to 701,000 µg/kg, or 89.79 percent removal
  - Total xylenes were reduced from an average of 2,203,333 to 633,333 µg/kg, or 71.26 percent removal
  - Naphthalene was reduced from an average of 1,816,667 to 1,703,333 µg/kg, or 6.24 percent removal. Unlike BTX which possess significant vapor pressures, naphthalene exhibits low vapor. Naphthalene also has a boiling point of 218°C (compared to 80 °C for benzene) therefore its removal efficiency by thermal treatment at 90°C and 100°C was expected to be comparatively lower than the BTX suite which exhibit appreciable volatility and lower boiling points.
- HC material treated at 100°C
  - Benzene was reduced from an average of 33,100,000 to 390,000 µg/kg, or 99.82 percent removal
  - Toluene was reduced from an average of 6,866,667 to 443,333 µg/kg, or 93.54 percent removal
  - Total xylenes were reduced from an average of 2,203,333 to 288,333 µg/kg, or 86.91 percent removal
  - Naphthalene was reduced from an average of 1,816,667 to 620,000 µg/kg, or 65.87 percent removal

Figure 4-5

Figure 4-6

- VR material treated at 90°C
  - Benzene was reduced from an average of 21,100,000 to 190,333 µg/kg, or 99.10 percent removal
  - Toluene was reduced from an average of 6,306,667 to 377,000 µg/kg, or 94.02 percent removal
  - Total xylenes were reduced from an average of 1,886,667 to 544,667 µg/kg, or 71.13 percent removal
  - Naphthalene increased from an average of 3,410,000 to 4,456,667 µg/kg. Based on the initial concentration and starting mass of VR, approximately 8.7 grams of naphthalene was present in the box reactor. Given the post treatment VR mass and final concentration, approximately 7.1 grams of naphthalene remained after heating. The difference in naphthalene mass in the reactor before and after treatment suggests that a small fraction was removed through heating. As previously stated, naphthalene is far less volatile than BTX and since this test condition occurred well below its boiling point, significant removal was not expected. Overall naphthalene mass reduction measured 18.85%.
- VR material treated at 100°C
  - Benzene was reduced from an average of 21,100,000 to 30,600 µg/kg, or 99.85 percent removal
  - Toluene was reduced from an average of 6,306,667 to 154,333 µg/kg, or 97.55 percent removal
  - Total xylenes were reduced from an average of 1,886,667 to 253,667 µg/kg, or 86.55 percent removal
  - Naphthalene was reduced from an average of 3,410,000 to 2,413,333 µg/kg, or 29.23 percent removal

Specific conclusions developed through this study include:

- The condensate produced during the thermal treatment generally produced similar observations and pH results for both the 90°C and 100°C treatments for each impoundment material. During the ramp up to 90°C and 100°C temperatures, the condensate for both VR and HC were generally clear with an oily NAPL on the surface.
- Condensate production seemed more prevalent and longer lasting for the VR material than the HC material.
- The TCLP results for the 90°C treatment are below the RCRA hazardous waste criteria, with the exception of chromium in the HC 90°C material.
- The HC 100°C TCLP results for benzene, pyridine, and chromium exceed RCRA hazardous waste criteria.
  - Chromium can be attributed to corrosion of the box reactor and thermocouple during treatment
  - Additional treatment time is expected to reduce pyridine and benzene to concentrations that would pass a TCLP test.
- Staining on the side walls of the reactor were observed in the VR material for both the 90° and 100°C treatments. The staining indicates that the material swells during heating and then subsides as it cools or subsides as the temperature stabilizes. Once the VR cools, it forms a flat, smooth, glassy surface below the stained sidewalls. It is not known if the swelling occurs along the side walls, or the entire volume of material swells uniformly. Swelling for the VR material appears to be approximately 30 to 40 percent during heating, and an expansion of approximately 3 to 5 percent after cooling.
- Extensive corrosion occurred in the reactors during the thermal treatment. Rust was observed in both the VR and HC materials, reactors, and thermocouples.
- An increase in the trace metals iron, manganese, aluminum, nickel and chromium occurred as compared to the untreated materials. Potential sources of trace metals include, (1) metal alloy and copper thermocouples that showed significant signs of rusting and pitting after treatment, (2) carbon steel reactors that showed significant rusting after treatment, and (3) aluminum foil used in the VR material reactors. Note the aluminum foil was used during testing to help facilitate removal of the treated materials from the reactor following testing; however, the foil partially disintegrated during testing.

- Moisture was retained by the material even after 24 days of heating at 90°C and after 14 days of heating at 100°C. Free water was observed in the bottom of the reactor in the HC material at both 90°C and 100°C. More free water was observed in the HC 100°C reactor.
- The HC responds to heat treatment in both target temperatures by forming a thin bubbly surface. Upon cooling, this surface hardens in place forming a thin fragile "shell." The VR 90°C material produced a "matted" taffy behavior after treatment and cooling, whereas the VR 100°C material hardens into a stiff material when cooled.

4.3 Physical Properties Testing

Since physical characteristics will figure strongly in the identification and implementation of remedial strategies for the impoundment materials, a portion of the bench-scale studies focused specifically on assessing change in material properties as a function of temperature and sustained heating conditions.

In general, physical property testing methods are designed for solid matrices representative of soil. Despite the solid and semi-solid nature of HC and VR impoundment materials, respectively, these materials do not behave like soil. As such, the application of physical testing methods to the impoundment materials was challenging. More importantly, however, is the understanding that testing methods likely introduced bias to several of the physical tests proposed and completed in this study. Based on limitations posed by the materials studied, results for several tests defined within the work plan were rejected by the testing laboratory. Where feasible, the rationale for data rejection will be discussed. The following sections summarize testing observations and laboratory results compiled to characterize the physical properties of VR and HC materials from Impoundment 2.

4.3.1 Viscosity and Specific Gravity Evaluation

Viscosity and density of the Impoundment 2 material was evaluated in the laboratory over the range of temperatures typically encountered during in-situ thermal treatment operations. Testing was proposed for both VR and HC material at the following temperature conditions: 30°C, 50°C, 70°C, and 90°C. Upon heating, VR material readily liquefied to allow measurement for specific gravity and viscosity. Measurement of specific gravity and viscosity for HC material; however, was not feasible given its physical properties. Analysis results are presented in Table 4-24. Photographs of the VR materials at the different temperatures are located in Appendix B.

TABLE 4-24
Laboratory Treatability Studies Report
Results of Viscosity Testing of HC and VR Materials

Test Parameter	HC Material[1]	VR Material
Density: at 30˚C (PCF)[3]	NT[5]	65.9[2]
Density: at 50˚C (PCF)	NT	66.2[2]
Density: at 70˚C (PCF)	NT	65.3[2]
Density: at 90˚C(PCF)	NT	66[2]
Viscosity: at ambient 17˚C (cP)[4]	NT	10,850 to 35,150
Viscosity: at 90˚C (cP)	NT	1,732 to 1,832

Notes:
[1] The viscosity of HC materials could not be tested because the material did not melt during the test temperatures.
[2.] Rejected Value
[3.] (PCF) Pounds per Cubic Foot
[4.] (cP) = Centipoise
[5.] NT = Not Tested

The testing results indicated that heating did not appreciably change the density of VR material; however, during layered heating studies described in Section 4.4.1, the VR material was observed to rise and float on the water as

the temperature reached 70°C. In addition, the volume of the VR material increased by approximately three during the trial reactor studies. Therefore, the density of the material must have decreased during thermal treatment. Consequently the density data for the VR material has been rejected.

During viscosity testing of the VR material at temperatures between ambient and 90°C "channeling" of the spindle agitating the sample was observed. The channeling was caused due to the viscosity of the material being too great to allow the material to flow back into the area swept by the spindle during agitation of the sample. Measurements between 30 and 90 therefore, were rejected by the laboratory based on direct observation of material behavior in the test cell.

4.3.2 Physical Properties – Untreated Materials

Selected physical properties of untreated Impoundment material are summarized in Table 4-25. As previously noted, the chemical characteristics of both the VR and HC material hindered completion of selected physical tests proposed in the work plan. The test apparatus for hydraulic conductivity (HC only) and triaxial shear testing require sample placement in a cylindrical membrane. Although material (HC only) was successfully packed into the sleeve, vapor emissions from the sample inflated the membrane in several locations and prevented completion of both tests. In lieu of performing hydraulic conductivity analysis, Laboratory Vane Shear testing was performed. This test provides shear strength of the material at a phi angle of zero (0). The VR sample did not hold form long enough to complete the UCS test. Photographs of the UCS testing are depicted in Appendix B.

TABLE 4-25
Laboratory Treatability Studies Report
Physical Properties of Untreated Impoundment Materials

Test Parameter	HC Material	VR Material
Hydraulic Conductivity	VE[1]	NR[2]
Bulk Density (PCF)[3]	59.2	65.9
Solid Specific Gravity	1.2	*
C.U. Triaxial Test (Cu) Ksf	VE	VE
Vane Shear (TSF)[4]	0.03	0.05
UCS	0.0 psi	Test Failed
Particle Size Distribution	% Gravel = 0.0 % Sand = 62.5 % Silt = 15.0 % Clay = 22.5	NR

Notes:
1. VE = Volatile Expansion
2. NR = Not Reported
3. PCF = Pounds per cubic foot
4. TSF = Tons per square foot

4.3.3 Physical Properties – Thermally Treated Impoundment Materials

Compressive strength of thermally treated impoundment materials was measured to help evaluate change in physical properties following sustained exposure to elevated temperatures. The physical properties of treated impoundment material are summarized in Table 4-26. Physical properties testing for treated impoundment materials were evaluated in accordance with the work plan with two notable exceptions:

- Based on sample matrix characteristics and observed behavior of the impoundment material after heating, representative samples could not be remolded for UCS, hydraulic conductivity, and triaxial shear testing. Rather, samples were prepared explicitly for compressive strength testing by placing untreated impoundment material into a plastic cylinder mold; the cylinders were subsequently heated to the target treatment temperatures and held under isothermal conditions for the same duration as the box reactor studies

previously discussed. Due to the expansion of the VR material, additional material was placed in the mold top daily to ensure it remained full. After several days at treatment temperature, the expansion of material outside the cylinder molds ceased. After the designated treatment times, the samples were removed from the molds and tested.

- Laboratory vane shear testing was substituted in lieu of the triaxial shear testing, which could not be tested due to volatile expansion of the material following sample placement within the membrane sleeves used to isolate the sample from the lateral confining pressure conditions.

TABLE 4-26
Laboratory Treatability Studies Report
Physical Properties of Treated Impoundment Materials

Test Parameter	VR 90°C	HC 90°C	VR 100°C	HC 100°C
Bulk Density (PCF) [1]	69.4	77.9	69	85.7
Porosity (1)	NR[2]	32.6	NR	16.44
Water Content (%)	26.73	19.54	11.44	25.46
Solid Specific Gravity	NR	1.55	NR	1.31
U.U. Triaxial Test	No Results	No Results	No Results	No Results
Vane Shear (TSF)[3]	> 0.6	0.28	> 0.6	0.25
UCS (psi)[3]	4.4	38.4	9.3	80.1
Hydraulic Conductivity	Failed under own weight	No Results	Failed under own weight	No Results
Particle Size Distribution	NR	%Gravel=21.6 %Sand=50.3 %Silt=3.8 %Clay=24.4	NR	%Gravel= 33.8 %Sand=37.9 %Silt=4.3 %Clay=24.0

Notes:
[1.] PCF = Pounds per cubic foot
[2.] NR = Not Reported
[3.] psi = pounds per square inch

Detailed study of physical properties of impoundment materials before and after thermal treatment provided the following observations:

- The density of the material did not significantly change with increasing temperature according to laboratory measurements completed. However, observations from the layering study discussed in the next section suggest that temperature does induce density reduction in both the VR and HC materials. This was documented when VR and HC both floated in a water column which was heated above approximately 80°C.
- The VR material did not have enough strength to maintain shape during triaxial testing. The HC material maintained a marginal shape during testing, but volatilization continued to produce vapor into the system (water lines) and prohibited performing the test.
- The laboratory vane shear testing indicated a significant increase in shear strength compared to untreated conditions in both materials. The VR material hardens into a stiff taffy and possesses greater shear strength than the HC material.
- Treated VR material possesses very little compressive strength (4 to 9 psi). With increasing load, treated VR material compressed and laterally expanded; the specimen did not shear of mechanically fracture during testing.
- Treated HC material appears to gain significant compressive strength achieving fracture points of 38 psi to 80 psi in the two conditions tested.

- The HC and VR treated at 100°C gained higher compressive strength values. Untreated unconfined strength for both materials was 0 psi.
- Particle size distribution testing revealed that the treated HC material has significantly more gravel size fractions than the untreated material. The treated material appears to produce conglomerates from smaller fractions. However, these larger fractions could be mechanically pulverized into smaller fractions.

4.4 Thermal Stability Testing

Several qualitative experiments were commissioned during the treatability study to gain basic knowledge of the effects of heating on Impoundment 2 materials.

4.4.1 Layered Heating

To investigate the vertical stability of water, VR, and HC during heating, a layered column was constructed using untreated materials and introduced to a temperature-controlled water bath. To emulate field conditions HC was placed beneath VR in the column; water was then added to cover the materials in the column The column was slowly heated to (30°C, 50°C, 70°C, and 90°C) and was allowed to remain at each temperature for 24 hours. During heating, column headspace was monitored for VOCs using a hand-held FID. The evaluation was performed to assess if natural mixing of the VR and HC materials would occur during heating. The following table (Table 4-27) provides a summary of the observations during heating of the layered materials.

TABLE 4-27
Laboratory Treatability Studies Report
Layered Heating Evaluation Laboratory Observations

Target Temperature	Description
30°C	The untreated material was heated at the target temperature for 24 hours. A gray layer formed on top of the VR sample. The VR, HC, and water did not move or mix throughout the test. Small peaks formed on the surface of the VR, but did not float upward. VOC range: 301.8 to 3859 ppm
50°C	The material was heated at the target temperature for 24 hours. A layer of the VR floated to the top of the water and remained there. The VR, HC, and water did not move or mix throughout the test. VOC range: 311.5 to 1544 ppm
70°C	The material was heated at the target temperature for 24 hours. A thin layer of the VR moved to the top of the water. The VR and HC material moved slightly up, and a thin layer of water formed beneath the HC (See photograph in Appendix B). Overnight, the water on top of the sample evaporated and the VR encapsulated around the HC. VOC range: 114.9 to 1331 ppm
90°C	The material was heated at the target temperature for 24 hours. The HC and VR material moved up and down the vial, exchanging places with the water. Overnight, the water on the sample evaporated and the VR expanded out of the vial. The materials did not mix. VOC range: 31 to above 50,000 ppm

4.4.2 Sample Reheating and Liquefaction Testing

To evaluate reversibility of heating operations and evaluate if heated VR material exhibited potential vertical mobility, the treated VR and HC from 100°C trials were reheated in a water bath at prescribed heating intervals and observed for 24 hours. Observations are summarized as follows:

- **HC 100**°C **-** The sample was prepared using a clear 40-milliliter vial. 1.4 inches of HC 100°C treated material and 1.4 inches of glass beads were inside. For 90 minutes the vial was suspended in a boiling water bath. The reheated material did not move down into glass beads, and liquefaction of the treated HC material did not occur.
- **VR 100**°C **-** The sample was prepared using a clear 40-milliliter vial 1.4 inches of VR 100°C treated material and 1.4 inches of glass beads were inside. For 90 minutes the vial was suspended in a boiling water bath. At a sample temperature of 65.5°C, migration of reheated VR material into the dry glass beads was observed. With increasing time, reheated VR material continued slow migration into the glass beads.

SECTION 5

Mixing, pH Adjustment, De-emulsification and Solidification/Stabilization Study and Results

5.1 Mixing, pH Adjustment, De-emulsification and Solidification/Stabilization Studies

The mixing, pH adjustment, de-emulsification and solidification/stabilization treatability studies evaluated different buffering and pozzolan reagents for both the thermally treated and raw impoundment material. The laboratory studies evaluated the performance of six different alkaline compounds and seven different pozzolan recipes (although not every compound screened is a pozzolan by definition, for this report all compounds screened are being referred to as pozzolans) for solidification/stabilization and was completed in four Tier levels of evaluations:

- Tier I – pH adjustment of homogenized impoundment materials and initial pozzolan screening
- Tier II – Pozzolan optimization based on results of Tier I screening
- Tier III – Secondary pozzolan addition of initially solidified/stabilized impoundment materials
- Tier IV – Supplemental solidification/stabilization testing to optimize permeability reduction and evaluate the effect upon the VOC fractions with the addition of powdered activated carbon (PAC) to the pozzolan mixture.

5.2 Bulk Heating

Bulk heating operations were conducted for the sole purpose of preparing materials for subsequent study in pH adjustment and solidification/stabilization evaluations. As such, detailed analytical evaluation of materials heated in each round was not performed. Samples of Impoundment 1 and 2 materials were heated in a temperature-controlled laboratory oven to simulate the effects of thermal treatment. Following a fixed heating period of 100^{o}C for 7 days, impoundment materials were cooled, visually inspected, and shipped to Remedial Construction Services, L.P. (RECON), for inclusion in their homogenization, pH adjustment and stabilization/solidification evaluations. The types and quantities of impoundment materials prepared were based on requirement for RECON to implement homogenization, pH adjustment, de-emulsification and solidification/stabilization treatability studies. Further discussion of these studies is found in Section 5. The impoundment materials were heated in three discrete heating rounds by Kemron and shipped for further study by RECON:

- Round 1 – Impoundment 2 HC and VR Material
- Round 2 – Impoundment 1 HC Material
- Round 3 – Impoundment 1 CA, CL, SSL, and VR materials.

During the bulk thermal heating, the impoundment materials were heated in Teflon-lined, 5-gallon steel buckets. A photograph of the bulk heating set up is depicted in Appendix B. Headspace above the impoundment materials was purged with air during the heating process and monitored using a flame ionization detector (FID) and a hydrogen sulfide and sulfur dioxide meter. Vapor generated during heating operations was passed through a surface condenser, a caustic impinger, and granulated activated carbon prior to discharge; vapor treatment operations were all conducted within a laboratory fume hood. Basic observations of bulk heating operations including vapor screening results, sample weight, and visual characteristics of impoundment materials pre- and post-heating were recorded. Results and observations compiled during bulk treatment are documented in Appendix C.

Because different types of impoundment materials were heated, experimental observations differed slightly between each round of heating. All heating rounds produced a significant volume of liquid condensate, which was generally composed of three distinct layers. The pH of the condensate was measured using pH paper and ranged from 2 to 10. Generally the condensate consisted of the following:

- An upper clear yellow layer of non-aqueous phase liquid (NAPL) with a strong gasoline-like odor; layer pH varied between material heated but typically ranged from 2-4 standard units (SU)
- A middle layer of white milky NAPL with a strong tar odor. The pH of this layer ranged from 2-3 SU.
- A bottom layer of light-green to grayish brown residue with a gritty texture. In general pH for this layer ranged from 8-10 SU.

Photographs, contained in Appendix B clearly illustrate the layers of condensate produced during bulk heating operations. Detailed physical descriptions of the bulk thermally heated materials are contained in Section 5 of this report.

5.3 Study Design and Treatability Objectives

The first step entailed different mixing/homogenization methodologies of thermally treated and raw impoundment materials, in-situ stabilization/solidification, and an initial 7-day cure-out followed by secondary ex-situ solidification/stabilization. The acidity of the impoundment materials has a detrimental effect upon the chemistry of the stabilization/solidification admixture. Therefore, the treatability testing included the addition of a pH buffering agent at the time of mixing and homogenization to raise the material pH to enable the stabilization/solidification admixture to optimally react and provide maximum final material strength and permeability reduction. Following the homogenization and pH adjustment, the solidification aspect of the process involves adding binders to absorb free porewater and liquids and increase the bearing capacity of the material. The stabilization part of the process involves adding chemicals to react with the impoundment material constituents to form a crystalline structure that reduces mobility of impoundment material compounds. Solidification/stabilization treatment typically accomplishes:

- Reduced contaminant solubility by formation of sorbed species or insoluble precipitates
- Improved physical strength and handling characteristics
- Decreased exposed surface area across which mass transfer of contaminants may occur
- Reduced contact between transport fluids and contaminants by reducing the permeability of the material

This work evaluated the benefits and feasibility of sequentially treating impoundment contents.

5.3.1 Homogenization

Impoundments 1 and 2 contain layers of various materials with physical properties that differ significantly from each other and between each impoundment. During a full-scale in-situ solidification/stabilization treatment scenario, the various material layers would be mixed together within the impoundment. To mimic this layered-impoundment structure at laboratory scale, 18-kg layered waste material models of each impoundment were created. Thermally treated impoundment materials were provided for testing as described in Section 5.2 of this Report. The target mass of 18 kg was used to create a model that fit into a standard 5-gallon polyethylene bucket. The mass of each layer in the model was based on data collected as part of the 2010 impoundment materials characterization (OBG 2010). Four 18-kg models were created, as follows:

- Impoundment 1 Raw Material (IMP 1 RM),
- Impoundment 2 Raw Material (IMP 2 RM)
- Impoundment 1 Thermally Treated Material (IMP 1 TT)
- Impoundment 2 Thermally Treated Material (IMP 2 TT)

Raw Impoundment Material

The impoundment materials (VR, HC, CA, SSL, and CL) collected in January and February 2012 were segregated in to individual containers by material type and shipped to RECON. Prior to constructing the models of each impoundment, the individual impoundment materials were mixed to create a more uniform blend for that material. The mixed individual impoundment materials were used to create models IMP 1 RM and IMP 2 RM. The target percentages used for the models were:

- Impoundment 1 (Layered Bottom to Top – see Figure 5-1)
  - HC Material – 28 percent
  - CA – 21 percent
  - SSL - 8 percent
  - HC Material - 28 percent
  - VR Material - 4.0 percent
  - CL Material -11 percent
  - One inch of Impoundment 1 water (water cap)
- Impoundment 2 (Layered Bottom to Top – see Figure 5-2)
  - HC – 43 percent
  - VR/HC 50/50 Blend – 14 percent
  - VR – 36 percent
  - VR/HC 50/50 Blend 7 percent
  - 1 inch of Impoundment 2 water (water cap)

In-situ solidification/stabilization treatment is often accomplished by advancing an auger through the contaminated media (column mixing) to mix solidification/stabilization additives into the media. Upon completion of the raw impoundment material models, auger-column mixing of the layers was simulated while sampling the headspace air to evaluate the emissions during this step. An apparatus was created to simulate column mixing within a sealed system. The apparatus consisted of a flat-disk drywall compound mixing blade with a shaft through a gasket-sealed aperture that could be advanced through the 18-kg model.

To support the characterization of the headspace above the impoundment materials, sampling ports were plumbed onto the lids of each sealed raw impoundment material model container (5-gallon bucket), which permitted sampling of the head space atmosphere above the model during column mixing (See Appendix B). During homogenization, the headspace was sampled for laboratory analysis for the following compounds:

- VOC and Naphthalene
- Aldehydes
- Malodorous Compounds
- Acid Gases

In addition to the samples collected for laboratory analysis, the headspace was screened for total VOCs, hydrogen sulfide and sulfur dioxide during homogenization using a FID, a hydrogen sulfide, and sulfur dioxide meter. Headspace sampling results during the column mixing of the raw impoundment material models are described in Section 5.4.1 of this Report.

Thermally Treated Impoundment Material

Column mixing and headspace sampling of the thermally treated (100^{o}C for 7 days) Impoundment 1 and 2 models was completed using the same methods that were employed for the raw material models with an exception that a 1-inch wood spade drill bit with an 18-inch shaft replaced the flat-disk drywall compound mixing blade used to homogenize the raw impoundment materials, as shown in Appendix B. This change was required to break up the hardened thermally treated impoundment materials. A water layer was not added to the top of the thermally treated impoundment material models because it is assumed that the water cover on the impoundments will be boiled off during a field-scale application of in-situ thermal heating.

Appendix B illustrates the Impoundment 1 thermally treated materials pre- and post-homogenization, and presents the Impoundment 2 thermally treated materials pre- and post- homogenization.

Figure 5-1

Figure 5-2

Headspace sampling was completed during the column mixing step and after column mixing; homogenization of the entire contents of the thermally treated models was attempted. This was unsuccessful for the Impoundment 1 model due to the "vitrified" or "sintered" physical properties of the thermally treated materials. Before homogenization, the top layer (CL) was chunky and the mixing blade was difficult to plunge into the materials. After homogenization, the various chunk-like layers remained intact.

Homogenization of the materials in the Impoundment 2 thermally treated model was also attempted; however, upon completion, the impoundment materials were not well mixed. Homogenization resulted in the chunks of Impoundment 2 material being disturbed by the mixing blade; however, the chunks of the individual impoundment materials appeared to remain intact. Headspace sampling was completed during column mixing of the Impoundment 2 model as well and sampling results during the column mixing of the thermally treated models are described in Section 5.4.1 of this Report.

During column mixing of the thermally treated impoundment 1 and 2 models, the headspace was sampled for laboratory analysis for the following compounds:

- VOC and Naphthalene
- Aldehydes
- Malodorous Compounds
- Headspace Acid Gases

As a result of the incomplete homogenization of the thermally treated impoundment models, it was concluded that a de-emulsification step was needed to successfully homogenize the thermally treated materials.

The de-emulsification procedure involved using the exothermic heat of hydration and chemistry of quicklime (HiCal Lime Kiln Dust [LKD]) to soften and breakdown the thermally treated impoundment materials. This de-emulsification procedure also combined the pH adjustment step with the homogenization step and resulted in the thermally treated models becoming pH adjusted. Headspace data from the de-emulsification step is located in Section 5.4.2.

5.3.2 pH Adjustment Material Assessment

Assessment of the effectiveness of pH adjustment compounds on the raw impoundment materials was conducted in two tiers. The first tier involved trial mixes of homogenized Impoundment 1 and 2 raw materials with six different pH adjustment compounds and measuring the pH of the completed mixtures. The second tier involved selecting the best performing pH adjusting compounds from the first tier to be utilized during the optimization studies within an enclosed glove box to allow for sampling the atmosphere created during the pH adjustment process. Only raw materials from Impoundments 1 and 2 were subjected to the pH adjustments because the conditions of the material after thermal treatment did not promote a direct pH adjustment without the application of a de-emulsification step.

Raw Impoundment Material

The pH changes resulting from increasing additions of six pH adjustment compounds were evaluated to determine the amount needed to raise the impoundment materials to a pH of 5 and the amount needed to reach a pH of greater than 8 SU but less than 11 SU. The six compounds evaluated as part of the pH adjustment testing were:

- Montague Hydrated Lime ($CaOH_2$) source from Montague, Michigan
- Carmeuse Hydrated Lime ($CaOH_2$) source sold by Carmeuse.
- Terrabond SC
- Lafarge Ravena Plant Reclaimed (Landfilled) Cement Kiln Dust (CKD)
- Sodium Hydroxide laboratory grade
- Mintek (Spent Lime)

Strength gain and headspace sampling results from the pH adjust screening are discussed in Section 5.4.2.

Thermally Treated Material

The pH adjustment step for the thermally treated impoundment materials was replaced by the application of an alkaline agent for the de-emulsification step. The addition of the HiCal LKD described in Section 5.3.1 for de-emulsification concurrently raised the pH of the thermally treated impoundment material to a pH of greater than 10 SU.

5.3.3 Pozzolan Screening

The objective of the pozzolan screening was to identify pozzolan amendment mixes for performance immediately after pozzolan addition. Screening was conducted using a pocket penetrometer to assess strength gain. It is to be noted that the use of the pocket penetrometer for initial strength gain is meant to be for comparative purposes only between amendment mixes. Pozzolans that achieved the greatest strength gain were carried through to optimization studies. Note that while the results of the performance screening is included in this report, longer-term performance monitoring of the pozzolans were conducted to evaluate strength gains as described in the work plan using 28- and 56-day Unconfined Compressive Strength testing procedures.

Tier I Screening – Raw and Thermally Treated Material

The Tier I pozzolan screening evaluated seven pozzolan mixture designs on their relative efficiencies for compressive strength gain using a pocket penetrometer. This assessment provides a general indication of compressive strength gain.

The following pozzolans were evaluated at a 30 percent addition in Tier I:

- PC: Portland Cement, Type I/II
- RECON Low Solids Stabilization (LSS) Blend A: Lafarge Portland Cement Type I/II 50 Percent and Circulating Fluidized Bed (CFB) Ash 50 Percent. CFB Ash was from Kimberly Clark and provided by HCF Construction/ Curt Forest
- RECON LSS Blend B: Lafarge Newcem 50 percent and Circulating Fluidized Bed Ash 50 Percent
- Terrabond/PC Blend: Terrabond-SC 50 percent and Buzzi Unicem Type I/II Portland Cement 50 Percent
- Newcem
- Newcem/PC Blend: Lafarge Newcem 75 percent and Lafarge Portland Cement Type I/II 25 percent
- Maxcem: Lafarge Maxcem (Lafarge's blend that includes 30 percent ground granulated blast furnace slag)

Homogenized samples of materials from the Impoundments 1 and 2 models were pH adjusted by an addition of Carmeuse Hydrated Lime immediately before the addition of pozzolans. Materials from the Impoundment 1 raw material models were pH adjusted by addition of 4 percent by weight Carmeuse Hydrated Lime, and homogenized Impoundment 2 raw materials were pH adjusted by addition of 6 percent Carmeuse Hydrated Lime. Samples of homogenized materials from both of the thermally treated models did not require pH adjustment because pH adjustment was completed during the de-emulsification step as described in Section 5.3.1. Strength data from the screening evaluations is presented in Section 5.4.3 of this report.

Tier II Optimization Assessment – Raw and Thermally Treated Material

Based on a review of the pocket penetrometer screening strength results of Tier 1 pozzolan screening, observation and professional judgment, the Tier II mix designs were selected for each of the four impoundment materials models (Impoundment 1 and 2 raw and thermally treated models).

TABLE 5-1
Laboratory Treatability Studies Report
Tier II Pozzolan Optimization Mix Designs

Optimization	Mix Composition
Impoundment 1 Raw Material Mix A	Carmeuse Hydrated Lime 4 percent by weight
	Lafarge Type I/II Portland Cement 7.5 percent by weight
	Omni Fluidized Bed Ash 7.5 percent by weight
Impoundment 1 Raw Material Mix B	Carmeuse Hydrated Lime 4 percent by weight
	Lafarge Newcem 10 percent by weight
Impoundment 2 Raw Material Mix A	Carmeuse Hydrated Lime 6 percent by weight (added as a slurry with 15 percent water by weight)
	Spent Fullers Earth 33 percent by weight
	Lafarge Newcem 15 percent by weight
	Omni Fluidized Bed Ash 15 percent by weight
Impoundment 2 Raw Material Mix B	Carmeuse Hydrated Lime 6 percent by weight (added as a slurry with 15 percent water by weight)
	Spent Fullers Earth 33 percent by weight
	Lafarge Newcem 30 percent by weight
Impoundment 1 Thermally Treated Mix A	Water 15 percent by weight
	Lafarge Type I/II Portland Cement 5 percent by weight
	Omni Fluidized Bed Ash 5 percent by weight
Impoundment 1 Thermally Treated Mix B	Water 15 percent by weight
	Lafarge Newcem 10 percent by weight
Impoundment 2 Thermally Treated Mix A	Water 15 percent by weight
	Lafarge Type I/II Portland Cement 5 percent by weight
	Omni Fluidized Bed Ash 5 percent by weight
Impoundment 2 Thermally Treated Mix B	Water 15 percent by weight
	Lafarge Newcem 10 percent by weight

These samples were homogenized and subject to headspace sampling during homogenization. Following initial curing, penetrometer-indicated strength testing was performed at 1 and 7 days along with headspace sampling conducted at the time of pozzolan addition and blending. Data from this portion of the study is located in Section 5.4.3 of this Report.

Tier III Optimization Assessment

As presented in the Treatability Testing Work Plan, a solidification/stabilization goal of greater than 15 psi UCS was initially evaluated for the solidification/stabilization testing. However, higher bearing capacities may need to be evaluated as part of the treatability. Therefore, the laboratory procedures for the Tier III Pozzolan Optimization testing were revised as follows:

- Following homogenization and pH buffering, Tier III consists of addition of approximately ½ of the pozzolan material to obtain a 48 to 72 hour early bearing capacity of greater than 10 psi (this would be sufficient for full-scale tracked equipment to bench out on top of this material to reach the next lift of impoundment material for pozzolan addition and blending operations).

- Following a 5- to 7-day cure time, the incompletely solidified/stabilized test material was removed from their sample mold, broken up to approximately ½-inch to ¾-inch fractions, and the remaining portion of the Tier III pozzolan and pulping water was added. This mixture was then remixed, and placed back into the sample mold for 28- and 56-day cure-out and testing.
  - This approach mimics the excavation of the initially and partially stabilized material and, secondary solidification/stabilization through a pugmill unit, for ultimate disposal.
- Following the 28-day cure-out period, pocket penetrometer, UCS strength, percent moisture, and TCLP and SPLP extraction testing were performed.
- Following the 56-day cure-out period, pocket penetrometer, UCS strength, percent moisture, and falling head permeability testing was performed.

5.4 Performance Results

The following sections provide the overall results of the homogenization, pH adjustment, and stabilization/solidification portions of the treatability testing.

5.4.1 Homogenization

Raw Impoundment Material

After the column mixing and headspace sampling the entire contents of the layered 18-kg models were completely homogenized using the same mixing tool used for the column mixing. The Impoundment 1 raw material model was the easiest to homogenize. Homogenization of raw Impoundment 1 materials resulted in a milkshake-like consistency. The water layer within became blended with the impoundment materials. No other additives were added during homogenization.

The material in the Impoundment 2 raw material model was somewhat blend-able. Before column mixing, the top-most layer (a VR/HC 50/50 blend) was rubbery to the extent that a depression of the mixing blade could be made and the impression held within the material. During homogenization, this top layer was observed to congeal and remain separate from the other material layers. It was difficult to mix this top layer into the other layers of impoundment material during homogenization. Layers beneath were not readily observable. The water layer on top of the model did not blend into the impoundment materials as occurred with Impoundment 1 raw material. No other additives were added during homogenization.

The analytical result from the headspace sampling of the raw impoundment materials during column mixing are in Table 5-2.

TABLE 5-2
Laboratory Treatability Studies Report
Selected Compounds Detected in the Headspace During Column Mixing of Raw Impoundment 1 and 2 Materials

Compound	IMP 1 RM Concentration (µg/m^3)	IMP 2 RM Concentration (µg/m^3)
	VOCs	
Benzene	5,200,000	20,000,000
Toluene	410,000	2,100,000
Naphthalene	< 25,000	< 120,000
Total Xylenes[2]	< 50,000	< 240,000
Ethylbenzene	< 25,000	< 120,000
Cumene	< 25,000	< 120,000
1,3,5-Trimethylbenzene	< 25,000	< 120,000
1,2,4-Trimethylbenzene	< 25,000	< 120,000
1,2-Dichlorobenzene	< 25,000	< 120,000

TABLE 5-2

Laboratory Treatability Studies Report

Selected Compounds Detected in the Headspace During Column Mixing of Raw Impoundment 1 and 2 Materials

Compound	IMP 1 RM Concentration (µg/m^3)	IMP 2 RM Concentration (µg/m^3)
Aldehydes		
Acetaldehyde	290[2]	20
Benzaldehyde	20	< 9
Butyraldehyde	30	< 10
Crotonaldehyde	< 10	< 10
Formaldehyde	< 10	< 10
Isovaleraldehyde	< 10	< 10
Propionaldehyde	90[2]	< 10
Valeradehyde	< 10	< 10
Acid Gases		
Sulfuric Acid	< 1,000	6,900
Malodorous Compounds		
Hydrogen Sulfide	4,900	230,000
Carbonyl Sulfide	88	260
Methyl Mercaptan	290	1,900
Dimethyl Sulfide	2,400	6,900
Carbon disulfide	24,000	220,000
Ethyl Methyl Sulfide	300	1,100
Thiophene	740	3,400
3-Methylthiophene	90	500

Notes:

[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.

[2.] Sorbent was saturated and result may be biased low.

Benzene and toluene were the VOCs detected in headspace samples during column mixing of the raw Impoundment 1 and 2 material models. Acid gases were not detected in the headspace above the Impoundment 1 model but were detected at 6,900 µg/m^3 in the headspace above the Impoundment 2 model. Concentrations of aldehydes were slightly higher in the headspace above the impoundment 1 model and concentrations of malodorous compounds were greater in the headspace above the Impoundment 2 model.

Thermally Treated Material

As indicated in Section 5.3.1, the thermally treated Impoundment 1 and 2 materials required a de-emulsification step to modify the physical properties of the materials so that homogenization could be completed. De-emulsification was first attempted on the model for Impoundment 1 and was suspended after temperatures of up to 140°F occurred and melted tar was observed. As a result of the high temperatures caused by the reaction of the HiCal LKD with the impoundment materials, the materials were transferred from the 5-gallon polyethylene bucket to a 20-gallon galvanized steel container. Photographs in Appendix B present the de-emulsification of Impoundment 1 thermally treated material with 30 percent addition of HiCal LKD and water. The Impoundment 2 thermal treatment model was also amended with a 30 percent addition of HiCal LKD and water. These amendments modified the physical properties of the thermally treated materials and allowed for thorough homogenization of the materials in the thermally treated models of Impoundment 1 and Impoundment 2.

The analytical results from the headspace sampling of the thermally treated impoundment materials during homogenization are in Table 5-3.

TABLE 5-3
Laboratory Treatability Studies Report
Selected Compounds Detected in the Headspace During Homogenization of Thermally Treated Impoundment 1 and 2 Materials

Compound	IMP 1 TT Concentration (µg/m³)	IMP 2 TT Concentration (µg/m³)
VOCs		
Benzene	530,000	530,000
Toluene	180,000	220,00
Naphthalene	< 3,400	< 3,100
Xylene (total)[1]	72,000	57,000
Ethylbenzene	5,200	5,000
Cumene	15,000	9,700
1,3,5-Trimethylbenzene	10,000	4,400
1,2,4-Trimethylbenzene	12,000	5,100
1,2-Dichlorobenzene	5,7000	6,600
Aldehydes		
Acetaldehyde	1,100	260[2]
Benzaldehyde	20	20
Butyraldehyde	80	30
Crotonaldehyde	< 10	< 10
Formaldehyde	< 10	< 10
Isovaleraldehyde	< 10	< 10
Propionaldehyde	80	70[2]
Valeradehyde	< 10	< 10
Acid Gases		
Sulfuric Acid	< 1,000	< 1,000
Malodorous Compounds		
Hydrogen Sulfide	5,400	180,000
Carbonyl Sulfide	850	42
Methyl Mercaptan	50	360
Dimethyl Sulfide	200	2,100
Carbon disulfide	7,400	4,500
Ethyl Methyl Sulfide	47	360
Thiophene	1,100	6,500
3-Methylthiophene	230	830
2-Ethylthiophene	210	1,500

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
[2.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.
ND = Not Detected.

The headspace analytical results for the homogenization of the thermally treated impoundment materials were similar to the data collected during the column mixing of the raw impoundment models, but the concentrations of constituents were lower. Benzene, toluene, and xylenes were the main VOCs detected in headspace samples during homogenization of the thermally treated Impoundment 1 and 2 material models. Acid gases were not detected in the headspace above either model. Concentrations of aldehydes were generally higher in the headspace above the impoundment 1 model and concentrations of malodorous compounds were generally greater (the exception being carbon sulfide) in the headspace above the Impoundment 2 model.

5.4.2 pH Adjustment Results

Raw Impoundment Material

Carmeuse Hydrated Lime was selected as the pH adjustment compound to be carried through to the Tier 2 pozzolan screening because the dosage rates were approximately one-half of other products tested. In addition, the lime can be applied as slurry, which was beneficial to modifying the physical properties of the thermally treated material to allow for homogenization.

The results of pH adjustment studies indicated that homogenized Impoundment 1 raw material would be treated with a 4 percent by weight of Carmeuse Hydrated Lime and homogenized Impoundment 2 raw material with a 6 percent by weight addition of Carmeuse Hydrated Lime to adjust the pH of the impoundment materials to a pH range of 11 SU for subsequent pozzolan screening. Headspace atmosphere at the final addition rate was sampled for laboratory analysis and the results are in Table 5-4.

TABLE 5-4
Laboratory Treatability Studies Report
Selected Headspace Compounds During pH Adjustment of Raw Impoundment 1 and 2 Materials

Compound	IMP 1 RM 4% $(CaOH)_2$ Concentration (µg/m³)	IMP 1 RM 4% HiCal LKD Concentration (µg/m³)	IMP 2 RM 6% $Ca(OH)_2$ Concentration (µg/m³)	IMP 2 RM 10% HiCal LKD Concentration (µg/m³)
VOCs				
Benzene	1,500,000	910,000	1,800,000	4,300,000
Toluene	140,000	130,000	19,000	550,000
Naphthalene	< 7,200	< 4,900	< 11,000	< 23,000
Xylene (total)[1]	8,800	8,500	< 11,000	34,000
Ethylbenzene	< 7,200	< 4,900	< 11,000	< 23,000
Cumene	< 7,200	< 4,900	< 11,000	< 23,000
1,3,5-Trimethylbenzene	< 7,200	< 4,900	< 11,000	< 23,000
1,2,4-Trimethylbenzene	< 7,200	< 4,900	< 11,000	< 23,000
1,2-Dichlorobenzene	< 1,400	< 980	< 2,200	< 4,500
Aldehydes				
Acetaldehyde	40	30	30	110
Benzaldehyde	< 9	< 9	< 9	20
Butyraldehyde	< 10	< 10	< 10	10
Crotonaldehyde	< 10	200[2]	< 10	< 10
Formaldehyde	< 10	10	10	< 10
Isovaleraldehyde	620[2]	530[2]	1,700[2]	2,800[2]
Propionaldehyde	< 10	< 10	< 10	< 10
Valeradehyde	< 10	< 10	< 10	< 10
Acid Gases				
Sulfuric Acid	< 1,000	< 1,000	< 1,000	< 1,000
Malodorous Compounds[3]				
Hydrogen Sulfide	NR	NR	NR	NR
Carbonyl Sulfide	NR	NR	NR	NR
Methyl Mercaptan	NR	NR	NR	NR

TABLE 5-4
Laboratory Treatability Studies Report
Selected Headspace Compounds During pH Adjustment of Raw Impoundment 1 and 2 Materials

Compound	IMP 1 RM 4% $(CaOH)_2$ Concentration (µg/m³)	IMP 1 RM 4% HiCal LKD Concentration (µg/m³)	IMP 2 RM 6% $Ca(OH)_2$ Concentration (µg/m³)	IMP 2 RM 10% HiCal LKD Concentration (µg/m³)
Dimethyl Sulfide	NR	NR	NR	NR
Carbon disulfide	NR	NR	NR	NR
Ethyl Methyl Sulfide	NR	NR	NR	NR
Thiophene	NR	NR	NR	NR
3-Methylthiophene	NR	NR	NR	NR
2-Ethylthiophene	NR	NR	NR	NR

Notes:
1. Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
2. Sorbent tube may have had breakthrough or possible migration. Results may be biased low.
3. Malodorous compounds Not Reported (NR) because samples were collected on the wrong sorbent tubes.

Headspace sampling during the pH adjustment studies of raw Impoundment 1 and 2 materials indicates that benzene and toluene are the most prevalent VOCs detected with lower concentrations of xylenes also being detected. Concentrations of VOCs and aldehydes detected in the atmosphere during pH adjustment were higher for Impoundment 2 materials than detected during pH adjustment of Impoundment 1 materials. Acid gases were not detected in the headspace for any of the samples collected during the pH adjustment. The concentrations of aldehydes were generally low, with isovaleraldehyde being detected at the highest concentration.

Penetrometer data collected after pH adjustment of the raw impoundment materials is presented in Table 5-5.

TABLE 5-5
Laboratory Treatability Studies Report
Tier I pH Adjustment and Penetrometer Screening Data

IMP and Mix Designation	pH alkaline buffering additive	1-day Cure Penetrometer Reading (psi)	7-day Cure Penetrometer Reading (psi)	28-day Cure Penetrometer Reading (psi)
IMP 1 RM $(CaOH)_2$	4% Carmeuse Hydrated Lime	0	0	0
IMP 1 RM HiCal LKD	30% addition of HiCal LKD and water	10.4	15.0	55.6
IMP 2 RM $(CaOH)_2$	6% Carmeuse Hydrated Lime	0	0	0
IMP 2 RM HiCal LKD	30% addition of HiCal LKD and water	34.7	33.7	31.2

The initial bearing capacity of the homogenized raw Impoundment 1 and 2 materials was zero and the addition of the alkaline buffering agent to the raw Impoundment 1 and 2 materials did not increase the material bearing capacity. However, the addition of the alkaline buffering/de-emulsification agents did significantly increasing the bearing capacity of the materials with up to 34.7 psi developing after one day of curing.

Thermally Treated Material

The pH adjustment step for the thermally treated impoundment materials was incorporated by the application of an alkaline agent for the de-emulsification step. The addition of the HiCal LKD described in Section 5.3.1 for de-emulsification concurrently raised the pH of the thermally treated impoundment material to a pH of greater than

10 SU. Headspace samples were collected during the de-emulsification step and the data are provided in Table 5-6.

TABLE 5-6
Laboratory Treatability Studies Report
Selected Headspace Compounds During De-Emulsification of Thermally Treated Impoundment 1 and 2 Materials

Compound	IMP 1 TT 30% (HiCal LKD & 30% H_2O) Concentration (µg/m^3)	IMP 2 RM (HiCal LKD & 30% H_2O) Concentration (µg/m^3)
VOCs		
Benzene	2,100	33,000
Toluene	2,100	40,000
Naphthalene	1,800	21,000
Xylene (total)[1]	2,830	25,500
Ethylbenzene	120	1,800
Cumene	520	4,500
1,3,5-Trimethylbenzene	580	4,200
1,2,4-Trimethylbenzene	850	6,300
1,2-Dichlorobenzene	940	26,000
Aldehydes		
Acetaldehyde	20	50
Benzaldehyde	< 9	< 9
Butyraldehyde	< 10	< 10
Crotonaldehyde	20	80[2]
Formaldehyde	< 10	< 10
Isovaleraldehyde	10	420[2]
Propionaldehyde	< 10	10
Valeradehyde	< 10	< 10
Acid Gases		
Sulfuric Acid	NA	< 1,000
Malodorous Compounds[3]		
Hydrogen Sulfide	240	7,200
Carbonyl Sulfide	13	19
Methyl Mercaptan	< 10	82
Dimethyl Sulfide	15	510
Carbon disulfide	22	190
Ethyl Methyl Sulfide	< 16	87
Thiophene	31	1,500
3-Methylthiophene	< 20	230
2-Ethylthiophene	< 23	430

TABLE 5-6
Laboratory Treatability Studies Report
Selected Headspace Compounds During De-Emulsification of Thermally Treated Impoundment 1 and 2 Materials

Compound	IMP 1 TT 30% (HiCal LKD & 30% H_2O) Concentration (µg/m^3)	IMP 2 RM (HiCal LKD & 30% H_2O) Concentration (µg/m^3)

Notes:
1. Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
2. Sorbent tube may have had breakthrough or possible migration. Results may be biased low.
NA = Not Available

The VOC content in the headspace during the de-emulsification of the thermally treated Impoundment materials was significantly lower than the VOC content in the headspace of the raw Impoundment materials. The major constituents continue to be benzene, toluene and xylenes: however, due to the lower method detection limits, other VOCs were also detected, including ethylbenzene, cumene, 1,3,5-trimethylbenzene, 1,2,4-trimethylbenzene and 1,2-dichlorobenzene. These compounds were also likely present in the headspace during the pH adjustment of the raw Impoundment materials but were not reported because of the higher reporting limits. Generally, the concentration of VOCs in the headspace during de-emulsification of thermally treated Impoundment 2 materials was an order of magnitude higher than the concentration of VOCs in the headspace during de-emulsification of thermally treated Impoundment 1 materials. Acid gases and aldehydes were also found at greater concentration is the headspace of the thermally treated impoundment 2 materials than in the headspace to the thermally treated Impoundment 1 materials during de-emulsification.

5.4.3 Pozzolan Screening

Raw Impoundment Material – Tier I Testing

For the Tier I small sample pozzolan recipe screening, the pocket penetrometer results indicated strengths at the 30 percent pozzolan addition rate for materials from the Impoundment 1 raw material model were higher than the initial target of 15 psi bearing capacity. Based on these results, the solidification agent addition rates for Impoundment 1 raw materials were reduced to 15 percent pozzolan addition in the subsequent optimization studies.

Penetrometer testing-indicated strengths for the materials from the Impoundment 2 raw materials model mix designs at 30 percent addition rates develops strengths slowly with strength of 0 psi after 28 days. Based on these results, spent Fuller's Earth was introduced as a binding agent in the some mix designs for the Impoundment 2 raw material model in the optimization studies.

Penetrometer test readings indicated strengths for the pH adjusted thermally treated impoundment materials from both impoundments with a 30 percent pozzolan addition of HiCal LKD were higher than the initial target of 15 psi bearing capacity. Based on these results, the addition rates of HiCal LKD were reduced to between 10 and 15 percent pozzolan addition in the subsequent optimization studies.

Raw Material Testing – Tier II Testing

Based on a review of the hand penetrometer screening strength results of Tier I Pozzolan Screening and professional judgment, the Tier II two-mix designs were selected for each of the Impoundment materials as described in Table 5-1. The Tier II pozzolan optimization penetrometer results for the cured raw Impoundment materials are shown in Table 5-7.

TABLE 5-7
Laboratory Treatability Studies Report
Tier II Pozzolan Raw Impoundment Material Optimization Study Penetrometer Results

Impoundment and Mix Designation	Batch Sheet Identification	1-day Cure Penetrometer Reading (psi)	3-day Cure Penetrometer Reading (psi)	7-day Cure Penetrometer Reading (psi)
IMP 1 RM Mix A	IMP 1 RM 003-C	48.6	>62.5	> 62.5
IMP 1 RM Mix B	IMP 1 RM 008-C	52.1	>62.5	> 62.5
IMP 2 RM Mix A	IMP 2 RM 004-C	10.4	27.8	59.0
IMP 2 RM Mix B	IMP 2 RM 008-C	3.47	24.3	59.0

Note:
62.5 psi is the maximum strength measurable on the penetrometer

Significant strength gains for raw Impoundment 1 and 2 materials were achieved during the Tier II optimization study. The Impoundment 2 material developed strength more slowly than Impoundment 1 material. However, after seven days, both the Impoundment 1 and 2 materials had developed similar strengths.

The strength data in Table 5-7 is representative of strength achieved after the pozzolans are mixed with the Impoundment material and allowed to cure. However, the cured solidified/stabilized Impoundment materials may require excavation from the impoundments after initial in-situ mixing. Therefore, to evaluate the strength of the excavated ISS treated Impoundment materials, the solidified/stabilized Impoundment materials were removed from their models and crushed after 5 to 7 days to simulate excavation, secondary pozzolan reagents and supplemental water were blended with the crushed samples which were then remolded to simulate placement in a landfill. Following an additional 28 days of curing for the cured materials were tested for UCS using ASTM D1633. The data from the excavation simulation (remixing) are located in Table 5-8. The laboratory data forms for the UCS data are located in Appendix D.

TABLE 5-8
Laboratory Treatability Studies Report
Tier II Pozzolan Raw Impoundment Material Remix UCS Results

Batch Sheet Identification	Batch Sheet Identification	28-Day Peak Load (lbs)	28-Day Peak Stress (psi)
IMP 1 RM 003-B	4% CL/7.5% LPC/7.5% HCF CFB	95	30.2
IMP 1 RM 004-B	4% CL/7.5% LN/7.5% HCF CFB	51	16.2
IMP 1 RM 005-B	4% CL/7.5% Terrabond/7.5% Buzzi PC	112	35.7
IMP 1 RM 006-B	4% CL/11.25% LN/3.75% LPC	71	22.6
IMP 1 RM 007-B	4% CL/15% LM	0	0
IMP 1 RM 008-B	4% CL/15% LN	53	16.9
IMP 2 RM 003-B	6%CL/15%H_2O/33%SFE/15% LPC/15% Omni FBC	50	15.9
IMP 2 RM 004-B	6%CL/15%H_2O/33%SFE/15% LN/15% Omni FBC	65	20.7
IMP 2 RM 005-B	6%CL/15%H2O/33%SFE/15% Terrabond/15% Buzzi PC	76	24.2
IMP 2 RM 006-B	6%CL/15%H2O/33%SFE/22.5% LN/15% LPC	58	18.5
IMP 2 RM 007-B	6%CL/15%H2O/33%SFE/30% LM	60	19.1
IMP 2 RM 008-B	6%CL/15%H2O/33%SFE/30% LN	65	20.7

TABLE 5-8
Laboratory Treatability Studies Report
Tier II Pozzolan Raw Impoundment Material Remix UCS Results

Batch Sheet Identification	Batch Sheet Identification	28-Day Peak Load (lbs)	28-Day Peak Stress (psi)

Note:
LM = LaFarge MaxCem
LN = LaFarge NewCem
CL = Carmeuse Lime
SFE = Spent Fullers Earth
FBC = Fluidized Bed Combustion Ash
LPC = LaFarge Portland Cement
PC = Portland Cement
H_2O = Water

The UCS results of the simulated excavation indicate limited strength will be developed after the solidified/stabilized raw Impoundment materials are excavated from the impoundments. The highest post removal strength (35.7 psi) for the mixed raw Impoundment 1 materials was achieved using 4% Carmeuse Lime/7.5% Terrabond/7.5% Buzzi Portland Cement mixture. The remixed impoundment 2 materials achieved lower strengths than the Impoundment 1 materials. The greatest post excavation strength developed (24.2 psi) for the treated Impoundment 2 materials was achieved using 6% Carmeuse Lime/15% water/33% Spent Fullers Earth/15% Terrabond/15% Buzzi Portland Cement blend. As a result of the low UCS achieved, an additional mixing step was added (Tier III) to achieve higher strengths after the materials are removed from the impoundments.

The head space above the mixing bowl was sampled and analyzed for VOCs, aldehydes and acid gases during the Tier II optimization studies. The recipes tested to solidify/stabilize the raw Impoundment materials are identified in Table 5-1. The results of the head space sampling are presented in Table 5-9. The headspace air analytical data is located in Appendix E.

TABLE 5-9
Laboratory Treatability Studies Report
Tier II Selected CoCs in Headspace During Mixing for Pozzolan Optimization Studies of Raw Materials

Compound	IMP 1 RM Mix A Concentration (µg/m³)	IMP 1 RM Mix B Concentration (µg/m³)	IMP 2 RM Mix A Concentration (µg/m³)	IMP 2 RM Mix B Concentration (µg/m³)
VOCs				
Benzene	120	330,000	800,000	85,000
Toluene	32	< 1,500	85,000	13,000
Naphthalene	< 1.8	< 1,500	< 8,200	< 440
Xylene (total)[1]	5	< 2,900	< 4,100	1,700
Ethylbenzene	< 1.8	< 1,500	< 4,100	< 440
Cumene	< 1.8	< 1,500	< 4,100	< 440
1,3,5-Trimethylbenzene	< 1.8	< 1,500	< 4,100	< 440
1,2,4-Trimethylbenzene	< 1.8	< 1,500	< 4,100	< 440
1,2-Dichlorobenzene	< 1.8	< 1,500	< 4,100	< 440
Aldehydes				
Acetaldehyde	260	260	200	220
Benzaldehyde	< 7	< 7	10	< 7
Butyraldehyde	20	20	20	40[2]
Crotonaldehyde	< 8	< 8	< 8	< 8
Formaldehyde	10	20	20	20

TABLE 5-9
Laboratory Treatability Studies Report
Tier II Selected CoCs in Headspace During Mixing for Pozzolan Optimization Studies of Raw Materials

Compound	IMP 1 RM Mix A Concentration (µg/m³)	IMP 1 RM Mix B Concentration (µg/m³)	IMP 2 RM Mix A Concentration (µg/m³)	IMP 2 RM Mix B Concentration (µg/m³)
Isovaleraldehyde	1,300[2]	1,200[2]	1,200[2]	1.800[2]
Propionaldehyde	10[2]	20	30[2]	20
Valeradehyde	< 7	< 7	< 7	< 7
Acid Gases				
Sulfuric Acid	< 800	< 800	< 800	< 800
Malodorous Compounds				
Hydrogen Sulfide	< 7	250	67	130
Carbonyl Sulfide	13	< 12	< 12	< 12
Methyl Mercaptan	< 10	< 10	< 10	< 10
Dimethyl Sulfide	86	41	96	110
Carbon disulfide	< 8	60	2,500	2,400
Ethyl Methyl Sulfide	< 16	< 16	< 16	< 16
Thiophene	39	23	55	69
3-Methylthiophene	< 20	< 20	< 20	< 20
2-Ethylthiophene	< 23	< 23	< 23	< 23

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
[2.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.

Concentrations of benzene, toluene and total xylenes were consistently detected in the glove box atmosphere during the pozzolan optimization mixing. The VOC results from IMP 1RM Mix A are believe to be biased low and the VOC data is not being considered valid. A potential cause for this is a depressurized Summa canister, which would collect a smaller volume of sample, resulting in the low bias. Acid gases were not detected in the glove box head space during pozzolan optimization mixing. Higher concentrations of malodorous compounds were detected in the headspace during the pozzolan optimization mixing of Impoundment 2 materials.

Thermally Treated Material

As previously stated, the pH adjustment step for the thermally treated impoundment materials was replaced by the application of an alkaline agent for de-emulsification. The addition of the HiCal LKD described in Section 5.3.1 for de-emulsification concurrently raised the pH of the thermally treated impoundment 1 and 2 materials to a pH of greater than 10 SU. Results from the in place strength pocket penetrometer evaluation for the pozzolan optimization of the thermally treated materials are shown in Table 5-10.

TABLE 5-10
Laboratory Treatability Studies Report
Tier II Thermally Treated Impoundment Material Pozzolan Optimization Study Penetrometer Results

Impoundment and Mix Designation	Batch Sheet Identification	1-day Cure Penetrometer Reading (psi)	3-day Cure Penetrometer Reading (psi)	7-day Cure Penetrometer Reading (psi)
IMP 1 TT Mix A	IMP 1 TT 004-C	> 62.5	> 62.5	> 62.5
IMP 1 TT Mix B	IMP 1 TT 008-C	> 62.5	> 62.5	> 62.5
IMP 2 TT Mix A	IMP 2 TT 003-C	41.7	> 62.5	> 62.5
IMP 2 TT Mix B	IMP 2 TT 008-C	62.5	> 62.5	> 62.5

TABLE 5-10
Laboratory Treatability Studies Report
Tier II Thermally Treated Impoundment Material Pozzolan Optimization Study Penetrometer Results

Impoundment and Mix Designation	Batch Sheet Identification	1-day Cure Penetrometer Reading (psi)	3-day Cure Penetrometer Reading (psi)	7-day Cure Penetrometer Reading (psi)

Note:
62.5 psi is the maximum strength measurable on the penetrometer

If the Impoundment materials are thermally treated prior to solidification/stabilization, they may be removed from the impoundments after ISS. Therefore, an evaluation of the materials strength after a simulated excavation was performed by crushing the solidified/stabilized materials after a cure period of 5 to 7 days. The crushed materials were then remixed and remolded and allowed to cure for an additional 28 days to simulate placement in a landfill. After curing, the remixed cylinder was tested for UCS using ASTM Method D1633. The results of the remixed USC testing is found in Table 5-11.

TABLE 5-11
Laboratory Treatability Studies Report
Tier II Thermally Treated Impoundment Pozzolan Material Remixed UCS Results

Batch Sheet Identification	Batch Sheet Identification	28-Day Peak Load (lbs)	28-Day Peak Stress (psi)
IMP 1 TT 003-B	15% H_2O/7.5% LPC/7.5% Omni FBC	51	16.2
IMP 1 TT 004-B	15% H_2O/7.5% LN/7.5% Omni FBC	32	10.2
IMP 1 TT 005-B	15% H_2O/7.5% Terrabond/7.5% Buzzi PC	81	25.8
IMP 1 TT 006-B	15% H_2O/11.25% LN/3.75% LPC	0	0
IMP 1 TT 007-B	15% H_2O/15% LM	0	0
IMP 1 TT 008-B	15% H_2O/15% LN	0	0
IMP 2 TT 003-B	15% H_2O/7.5% LPC/7.5% Omni FBC	32	10.2
IMP 2 TT 004-B	15% H_2O/7.5% LN/7.5% Omni FBC	18	5.7
IMP 2 TT 005-B	15% H_2O/7.5% Terrabond/7.5% Buzzi PC	48	15.3
IMP 2 TT 006-B	15% H_2O/11.25% LN/3.75% LPC	39	12.4
IMP 2 T 007-B	15% H_2O/15% LM	22	7.0
IMP 2 TT 008-B	15% H_2O/15% LN	39	12.4

Note:
LM = LaFarge MaxCem
LN = LaFarge NewCem
CL = Carmeuse Lime
SFE = Spent Fullers Earth
FBC = Fluidized Bed Combustion Ash
LPC = LaFarge Portland Cement
PC = Portland Cement
H_2O = Water

The UCS results indicate that if the solidified/stabilized thermally treated materials are excavated from the impoundments, they will have minimal strength when placed at the final disposal location. The UCS ranged from none (sample crumbled when removed from the UCS mold) to 25.8 psi. The data suggest that a Tier III study for the thermally treated impoundment materials was required to evaluate methods to achieve post excavation strength.

During the Tier II pozzolan optimization studies, headspace samples were collected from the atmosphere inside the glove box where pozzolan mixing occurred and are reported in Table 5-12 for the thermally treated Impoundment materials.

TABLE 5-12
Laboratory Treatability Studies Report
Tier II Selected CoCs in Headspace During Mixing for Pozzolan Optimization Studies of Thermally Treated Impoundment Materials

Compound	IMP 1 TT Mix A Concentration (µg/m^3)	IMP 1 TT Mix B Concentration (µg/m^3)	IMP 2 TT Mix A Concentration (µg/m^3)	IMP 2 TT Mix B Concentration (µg/m^3)
VOCs				
Benzene	5,300	5,500	6,000	4,300
Toluene	1,300	1,300	3,500	2,500
Naphthalene	99	130	< 35	500
Xylene (total)[1]	323	449	1,520	1,230
Ethylbenzene	< 28	< 30	100	79
Cumene	46	65	220	170
1,3,5-Trimethylbenzene	36	56	140	140
1,2,4-Trimethylbenzene	52	91	190	230
1,2-Dichlorobenzene	120	170	660	840
Aldehydes				
Acetaldehyde	130[2]	60	40	77[2]
Benzaldehyde	10	10	20	20
Butyraldehyde	10	10	20	10
Crotonaldehyde	< 8	< 8	< 8	< 8
Formaldehyde	< 7	8	< 7	< 7
Isovaleraldehyde	410[2]	140[2]	370[2]	380[2]
Propionaldehyde	20	9	10	< 7
Valeradehyde	< 7	< 7	< 7	< 7
Acid Gases				
Sulfuric Acid	< 800	< 800	< 800	< 800
Malodorous Compounds				
Hydrogen Sulfide	< 7	< 7	< 7	< 7
Carbonyl Sulfide	< 12	< 12	< 12	< 12
Methyl Mercaptan	< 10	< 10	< 10	< 10
Dimethyl Sulfide	< 13	< 13	< 13	< 13
Carbon disulfide	25	16	60	46
Ethyl Methyl Sulfide	< 16	< 16	< 16	< 16
Thiophene	< 17	< 17	55	59
3-Methylthiophene	< 20	< 20	< 20	< 20
2-Ethylthiophene	< 23	< 23	< 23	< 23

TABLE 5-12
Laboratory Treatability Studies Report
Tier II Selected CoCs in Headspace During Mixing for Pozzolan Optimization Studies of Thermally Treated Impoundment Materials

Compound	IMP 1 TT Mix A Concentration (µg/m^3)	IMP 1 TT Mix B Concentration (µg/m^3)	IMP 2 TT Mix A Concentration (µg/m^3)	IMP 2 TT Mix B Concentration (µg/m^3)

Notes:
[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
[2.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.

Concentrations of benzene in the headspace of the glove box from the Tier II pozzolan mixing of the thermally treated Impoundment materials were approximately two orders of magnitude lower than concentration detected during the pozzolan mixing of the raw impoundment materials. In addition, the concentrations of aldehydes decreased in the glove box atmosphere and the number and concentration of malodorous compounds also were significantly lower as compared to the raw impoundment material following pozzolan mixing.

5.4.4 Tier III Post Excavation Evaluation

Results of the Tier II studies indicated that after mixing pozzolans with the Impoundment materials and excavation of the materials, additional strength may be required to increase the number of disposal options. The Tier III studies evaluated the results of a second ex situ mixing step to increase the post removal strength of the impoundment materials.

Raw Impoundment Materials

For the Tier III studies, the raw Impoundment materials were initially mixed with the pozzolan blends outlined in Table 5-1 and packed into UCS cylinders to cure for 5 to 7 days. After curing the samples were removed from the cylinders, crushed and remixed with a slurry of 10% LaFarge Portland Cement and 8% water and repacked into UCS cylinders to cure for 28 and 56 days. The results of the 28 and 56 day UCS tests are provided in Table 5-13.

TABLE 5-13
Laboratory Treatability Studies Report
Tier III Raw Impoundment Material Pozzolan Remix UCS Results

Impoundment and Mix Designation	Batch Sheet Identification	28-Day Peak Load (lbs)	28-Day Peak Stress (psi)	56-Day Peak Load (lbs)	56-Day Peak Stress (psi)
IMP 1 RM Mix A	IMP 1 RM 003-C	170	54.1	218	69.4
IMP 1 RM Mix B	IMP 1 RM 008-C	282	89.8	290	92.3
IMP 2 RM Mix A	IMP 2 RM 004-C	134	42.7	198	63.0
IMP 2 RM Mix B	IMP 2 RM 008-C	162	51.6	180	57.3

The results of the Tier III remixed UCS testing indicate significant strength gains over the Tier II remixing results. The remixed solidified/stabilized raw Impoundment 1 materials achieved up to 92.3 psi and the remixed solidified/stabilized raw Impoundment 2 materials achieved up to 63.0 psi. These values would be sufficient for placement in most landfills. The strength increased in each sample from the 28 to the 56 day cure.

In addition, cylinders of remixed solidified/stabilized raw Impoundment materials were prepared for permeability testing. Similar to the UCS testing, the permeability cylinders were prepared using the initial mix recipes from Table 5-1 to prepare an initial cylinder of solidified/stabilized materials. The initial cylinders were crushed after 5 to 7 days of curing and remixed using a slurry of 10% LaFarge Portland Cement and 8% water. Then new cylinders were molded and allowed to cure for 28 days before evaluated for permeability using ASTM Method D5084.

Results of the permeability testing are located in Table 5-14. The permeability laboratory data forms are located in Appendix F.

TABLE 5-14
Laboratory Treatability Studies Report
Tier III Raw Impoundment Material Pozzolan Remix Permeability Results

Impoundment and Mix Designation	Batch Sheet Identification	Permeability (cm/sec)
IMP 1 RM Mix A	IMP 1 RM 003-C	8.07×10^{-5}
IMP 1 RM Mix B	IMP 1 RM 008-C	5.45×10^{-4}
IMP 2 RM Mix A	IMP 2 RM 004-C	2.91×10^{-4}
IMP 2 RM Mix B	IMP 2 RM 008-C	1.63×10^{-3}

The permeability of the remixed solidified/stabilized Impoundment materials ranged from 1.63×10^{-3} cm/sec for raw Impoundment 2 material mixed with Mix B to 8.07×10^{-5} cm/sec for raw impoundment 1 material mixed with Mix A. The permeability of the Impoundment 1 material was slightly lower than the permeability achieved from the mixing of the Impoundment 2 materials.

In addition to the UCS and permeability testing, the remixed solidified/stabilized raw impoundment materials were analyzed using TCLP methods after 28 days of curing to evaluate chemical leaching from the materials if they were placed in a landfill. The results of the TCLP testing of the solidified Impoundment 1 and 2 materials is provided in Table 5-15.

TABLE 5-15
Laboratory Treatability Studies Report
Tier III Raw Impoundment Material for Pozzolan Remix TCLP Results

Compound	Regulatory Level (mg/L)	Raw IMP 1 (mg/L)	Raw IMP 2 (mg/L)	IMP 1 RM Mix A Concentration (mg/L)	IMP 1 RM Mix B Concentration (mg/L)	IMP 2 RM Mix A Concentration (mg/L)	IMP 2 RM Mix B Concentration (mg/L)
VOCs							
Vinyl Chloride	0.2	< 200	< 200	< 0.040	< 0.040	< 0.040	< 0.040
1,1-Dichloroethene	0.7	< 200	< 200	< 0.040	< 0.040	< 0.040	< 0.040
2-Butanone (MEK)	200	< 500	< 500	< 0.100	< 0.100	< 0.100	< 0.100
Chloroform	6.0	< 150	< 150	< 0.030	< 0.030	< 0.030	< 0.030
1,2-Dichloroethane	0.5	< 200	< 200	< 0.040	< 0.040	< 0.040	< 0.040
Carbon Tetrachloride	0.5	< 200	< 200	< 0.040	< 0.040	< 0.040	< 0.040
Benzene	0.5	434[a]	469[a]	15.4	14.2	90.3[a]	113.0[a]
Trichloroethene	0.5	<200	<200	< 0.040	< 0.040	< 0.040	< 0.040
Tetrachloroethene	0.7	< 200	< 200	< 0.040	< 0.040	< 0.040	< 0.040
Chlorobenzene	100	< 200	< 200	< 0.040	< 0.040	< 0.040	< 0.040
1,4-Dichlorobenzene	7.5	< 200	< 200	< 0.040	0.0492 J	0.105	0.114

[a] = Results from 2nd dilution of sample
J = Estimated value below the Method Reporting Limits (MRL)

Benzene was the only compound detected in the leachate during the TCLP analysis above the regulatory level. The 28-day TCLP data indicate that the concentration of benzene in the leachate from the solidified/stabilized impoundment materials ranged from 14.2 mg/L to 113 mg/L. The leachate benzene concentration from the raw impoundment material was 434 mg/L. The leachate benzene concentration reduction for the treated

Impoundment 1 materials was up to 96.7% and the leachate benzene concentration reduction for the treated Impoundment 2 materials was up to 80.7%. The solidified/stabilized Impoundment 2 materials leachate contained a significantly higher benzene concentration than the leachate from the solidified/stabilized Impoundment 1 materials. Each of the 4 pozzolan receipts tested could not reduce the leachate benzene concentration to below the regulatory level, and the solidified/stabilized Impoundment 1 and 2 materials would be considered hazardous per RCRA regulations. Detection limits for other VOCs in the leachate of the raw impoundment materials were above their respected regulatory levels. None of these compounds were detected in the leachate of the solidified/stabilized impoundment materials above their respective regulatory levels.

In addition to the TCLP analysis, the remixed solidified/stabilized raw impoundment materials were also analyzed using SPLP methods after 28 days of curing, which simulates leaching from the Impoundment materials resulting from precipitation infiltration. The results of the SPLP analysis of the cured remixed solidified Impoundment 1 and 2 materials is provided in Table 5-16.

TABLE 5-16
Laboratory Treatability Studies Report
Tier III Raw Impoundment Material for Pozzolan Remix SPLP Results

Compound	TCLP Regulatory Level (mg/L)	Raw IMP 1 (mg/L)	Raw IMP 2 (mg/L)	IMP 1 RM Mix A Concentration (mg/L)	IMP 1 RM Mix B Concentration (mg/L)	IMP 2 RM Mix A Concentration (mg/L)	IMP 2 RM Mix B Concentration (mg/L)
				VOCs			
Vinyl Chloride	0.2	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
1,1-Dichloroethene	0.7	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
2-Butanone (MEK)	200	< 1,000	< 1,000	< 0.100	< 0.100	< 0.100	< 0.100
Chloroform	6.0	< 300	< 300	< 0.030	< 0.030	< 0.030	< 0.030
1,2-Dichloroethane	0.5	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
Carbon Tetrachloride	0.5	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
Benzene	0.5	403[a]	466[a]	17.4	14.3	93.6[a]	121[a]
Trichloroethene	0.5	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
Tetrachloroethene	0.7	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
Chlorobenzene	100	< 400	< 400	< 0.040	< 0.040	< 0.040	< 0.040
1,4-Dichlorobenzene	7.5	< 400	< 400	0.0447	0.0407 J	0.114	0.119

[a] = Results from 2nd dilution of sample
J = Estimated value below the Method Reporting Limits (MRL)

The SPLP data for the untreated Impoundment materials indicates that leaching was greater from Impoundment 2 materials and benzene was the only compound detected in the leached water above the TCLP regulatory level. Detection limits for each VOC in the leachate; except for benzene, exceeded their respective regulatory limits. The benzene concentration in the leachate from the SPLP testing was similar to the benzene concentration in the leachate from the TCLP testing. The leachate benzene reduction achieve by the solidification/stabilization and remixing of the raw Impoundment 1 materials was up to 96.5% and the leachate benzene reduction achieved by solidification/stabilization and remixing of the Impoundment 2 materials was up to 79.9%. The benzene concentration in the leached water from the Impoundment 2 materials was significantly greater than the benzene concentration in the leached water from the Impoundment 1 materials.

Air samples were collected and analyzed for VOCs, aldehydes and acid gases during the crushing and remixing of the initially solidified/stabilized impoundment materials. The samples were collected to evaluate emissions that may occur during excavation of the impoundment materials after an initial solidification/stabilization step. In addition, headspace samples were collected from the remixed impoundment materials UCS molds to evaluate off gassing occurring after 28 days of curing. The Tier III air sampling data is found in Table 5-17.

TABLE 5-17
Laboratory Treatability Studies Report
Tier III Raw Impoundment Material Selected CoCs in Headspace During Remixing and 28-Day Cure

	IMP 1 RM Mix A Concentration (µg/m³)		IMP 1 RM Mix B Concentration (µg/m³)		IMP 2 RM Mix A Concentration (µg/m³)		IMP 2 RM Mix B Concentration (µg/m³)	
Compound	**Remixing**	**Day 28**	**Remixing**	**Day 28**	**Remixing**	**Day 28**	**Remixing**	**Day 28**
VOCs								
Benzene	36,000,000	2,500,000	28,000,000	940,000	26,000,000	6,900,000	44,000,000	15,000,000
Toluene	2,900,000	360,000	2,100,000	310,000	2,100,000	860,000	3,600,000	1,600,000
Naphthalene	< 230,000	< 14,000	< 170,000	< 5,700	< 180,000	< 34,000	< 290,000	< 70,000
Xylene (total)[1]	< 450,000	< 29,000	< 350,000	34,500	< 360,000	< 67,000	< 580,000	< 140,000
Ethylbenzene	< 230,000	< 14,000	< 170,000	< 5,700	< 180,000	< 34,000	< 290,000	< 70,000
Cumene	< 230,000	< 14,000	< 170,000	< 5,700	< 180,000	< 34,000	< 290,000	< 70,000
1,3,5-Trimethylbenzene	< 230,000	< 14,000	< 170,000	< 5,700	< 180,000	< 34,000	< 290,000	< 70,000
1,2,4-Trimethylbenzene	< 230,000	< 14,000	< 170,000	< 5,700	< 180,000	< 34,000	< 290,000	< 70,000
1,2-Dichlorobenzene	< 230,000	< 14,000	< 170,000	< 5,700	< 180,000	< 34,000	< 290,000	< 70,000
Aldehydes								
Acetaldehyde	20	110	10	10	20	50	40	280
Benzaldehyde	< 9	40	< 9	50	< 9	9	< 9	40
Butyraldehyde	< 10	10	< 10	10	< 10	10	< 10	30
Crotonaldehyde	30	< 8	< 10	< 8	< 10	< 8	< 10	< 8
Formaldehyde	< 10	10	< 10	10	< 10	8	< 10	10
Isovaleraldehyde	190	< 7	230[2]	< 7	460[2]	< 7	470[2]	< 7
Propionaldehyde	< 10	< 7	< 10	8	< 10	< 7	< 10	20
Valeradehyde	< 10	< 7	< 10	< 7	< 10	< 7	< 10	< 7
Acid Gases								
Sulfuric Acid	< 1,000	< 80	< 1,000	< 80	< 1,000	< 80	< 1,000	< 80
Malodorous Compounds								
Hydrogen Sulfide	35	< 7	7	< 7	7	< 7	70	< 7
Carbonyl Sulfide	26	< 12	92	< 12	96	73	740	210
Methyl Mercaptan	< 10	< 10	< 10	< 10	< 10	< 10	< 98	< 10
Dimethyl Sulfide	760	< 13	870	< 13	1,200	1,200	3,400	1,600
Carbon disulfide	3,400	1,800	9,000	590	24,000	12,000	43,000	19,000
Ethyl Methyl Sulfide	210	16	210	< 16	220	150	550	230
Thiophene	660	300	800	83	1,100	780	2,400	2,200
3-Methylthiophene	150	48	170	< 20	150	160	270	330
2-Ethylthiophene	30	33	79	< 23	65	110	< 230	220

Notes:
1. Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.
2. Sorbent tube may have had breakthrough or possible migration. Results may be biased low.

Emissions during the remixing step were dominated by benzene and toluene. Other VOCs were not detected in the samples because the high concentration of benzene required sample dilution and raised the detection limits.

The highest concentration of VOCs were detected during the remixing step of the Impoundment 2 materials treated with Mix B. The headspace above the remixed solidified/stabilized impoundment 2 materials generally contained higher concentrations of aldehydes and malodorous compounds. The concentration of most compounds detected in the headspace above the solidified/stabilized remixed impoundment materials decreased after 28 days of curing as compared with the remixing step concentrations.

Thermally Treated Material

The thermally treated impoundment materials was subjected to the same testing as the solidified/stabilized impoundment materials. Based on low strengths achieved from the Tier II testing, the Tier III testing involved using the mixtures described in Table 5-1 for an initial solidification/stabilization followed by a remix using a slurry of 10% LaFarge Portland Cement and 8% water. Test cylinders were prepared after initial solidification of thermally treated Impoundment 1 and 2 materials and allowed to cure for 5 to 7 days. After curing, the materials were removed from the cylinders and crushed to simulated excavation from the impoundments. The crushed materials were remixed with the secondary pozzolan reagents and water and placed into new UCS cylinders and allowed to cure for either 28 or 56 days. At 28 and 56 days the cylinders were tested for USC following ASTM Method D1633. The results of the 28-day and 56-day USC tests are provided in Table 5-18.

TABLE 5-18
Laboratory Treatability Studies Report
Tier III Thermally Treated Impoundment Material Pozzolan Remix UCS Results

Impoundment and Mix Designation	Batch Sheet Identification	28-Day Peak Load (lbs)	28-Day Peak Stress (psi)	56-Day Peak Load (lbs)	56-Day Peak Stress (psi)
IMP 1 TT Mix A	IMP 1 TT 004-C	212	67.5	358	114
IMP 1 TT Mix B	IMP 1 TT 008-C	275	87.5	189	60.2
IMP 2 TT Mix A	IMP 2 TT 003-C	183	58.3	199	63.3
IMP 2 TT Mix B	IMP 2 TT 008-C	151	48.1	144	45.8

The results for the UCS of the solidified/stabilized thermally treated impoundment materials were similar to the UCS results for the solidified/stabilized impoundment materials. Significant strength gains were achieved during the remixing step. The thermally treated Impoundment 1 materials achieved better strength (peak strength of 114 psi) than the thermally treated Impoundment 2 materials (peak strength of 63.3 psi). As a result of the remixing, the impoundment materials achieved enough strength to be accepted at most landfill disposal facilities.

Additional cylinders of remixed solidified/stabilized thermally treated Impoundment materials were prepared for permeability testing. The cylinders were prepared using the mix recipes from Table 5-1 then remixed with the slurry of 10% LaFarge Portland Cement and 8% water. Each cylinder was allowed to cure for 28 days and then subjected to permeability testing using ASTM Method D5084. Results of the permeability testing are located in Table 5-19.

TABLE 5-19
Laboratory Treatability Studies Report
Tier III Thermally Treated Impoundment Material Pozzolan Remix Permeability Results

Impoundment and Mix Designation	Batch Sheet Identification	Permeability (cm/sec)
IMP 1 TT Mix A	IMP 1 TT 004-C	1.09×10^{-4}
IMP 1 TT Mix B	IMP 1 TT 008-C	1.14×10^{-4}
IMP 2 TT Mix A	IMP 2 TT 003-C	1.19×10^{-5}
IMP 2 TT Mix B	IMP 2 TT 008-C	8.46×10^{-4}

TABLE 5-19
Laboratory Treatability Studies Report
Tier III Thermally Treated Impoundment Material Pozzolan Remix Permeability Results

Impoundment and Mix Designation	Batch Sheet Identification	Permeability (cm/sec)

Note:

The permeability of the remixed solidified/stabilized Impoundment materials ranged from 8.46 x 10^{-4} cm/sec for thermally treated Impoundment 2 material mixed with Mix B to 1.19 x 10^{-5} cm/sec for thermally treated impoundment 2 material mixed with Mix A. The pozzolans tested with the thermally treated material achieved slightly better permeability than the raw impoundment materials.

The thermally treated impoundment materials were also tested for TCLP. The results of the TCLP testing of the solidified Impoundment 1 and 2 materials is provided in Table 5-20.

TABLE 5-20
Laboratory Treatability Studies Report
Tier III Thermally Treated Impoundment Material for Pozzolan Remix TCLP Results

Compound	Regulatory Level mg/L)	Thermally Treated IMP 1 (mg/L)	Thermally Treated IMP 2 (mg/L)	IMP 1 TT Mix A Concentration (mg/L)	IMP 1 TT Mix B Concentration (mg/L)	IMP 2 TT Mix A Concentration (mg/L)	IMP 2 TT Mix B Concentration (mg/L)
VOCs							
Vinyl Chloride	0.2	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
1,1-Dichloroethene	0.7	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
2-Butanone (MEK)	200	0.022	0.110	< 0.100	< 0.100	< 0.100	< 0.100
Chloroform	6.0	< 0.0015	< 0.003	< 0.030	< 0.030	< 0.030	< 0.030
1,2-Dichloroethane	0.5	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Carbon Tetrachloride	0.5	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Benzene	0.5	2.19[a]	6.66[a]	3.66	2.06	1.16	1.04
Trichloroethene	0.5	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Tetrachloroethene	0.7	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Chlorobenzene	100	0.0023 J	0.010	< 0.040	< 0.040	< 0.040	< 0.040
1,4-Dichlorobenzene	7.5	0.0189	0.109	< 0.040	< 0.040	0.096 J	0.0904 J

[a] = Results from 2nd dilution of sample
J = Estimated value below the Method Reporting Limits (MRL)

During the TCLP testing of the thermally treated impoundment materials, benzene was the only compound detected in the leachate above the regulatory level. Treatment of the thermally treated Impoundment materials did not reduce benzene leachability to below the regulatory level and Impoundment 1 materials leached more benzene than Impoundment 2 materials. The solidification/stabilization and remixing of the thermally treated Impoundment 1 materials reduced benzene leaching by up to 47.0% and the solidification/stabilization and remixing of the thermally treated Impoundment 2 materials reduced benzene leaching by up to 71.6%. The concentration of benzene in the leachate from materials treated with IMP 1 TT Mix A was greater than the concentration of benzene in the leachate from the thermally treated Impoundment 1 materials that were not solidified/stabilized. This may have resulted from incomplete homogenization of the thermally treated materials which as indicated in Section 5.3.1, due to difficulty homogenizing hardened large chunks of impoundment material from the bulk thermal treatment operations.

After 28 days of curing, the solidified/stabilized impoundment materials were analyzed for SPLP. The results of the SPLP testing for the thermally treated impoundment materials is found in Table 5-21.

TABLE 5-21
Laboratory Treatability Studies Report
Tier III Thermally Treated Impoundment Material for Pozzolan Remix SPLP Results

Compound	TCLP Regulatory Level (mg/L)	Thermally Treated IMP 1 (mg/L)	Thermally Treated IMP 2 (mg/L)	IMP 1 TT Mix A Concentration (mg/L)	IMP 1 TT Mix B Concentration (mg/L)	IMP 2 TT Mix A Concentration (mg/L)	IMP 2 TT Mix B Concentration (mg/L)
VOCs							
Vinyl Chloride	0.2	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
1,1-Dichloroethene	0.7	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
2-Butanone (MEK)	200	0.0528	0.110	< 0.100	< 0.100	< 0.100	< 0.100
Chloroform	6.0	< 0.0015	< 0.003	< 0.030	< 0.030	< 0.030	< 0.030
1,2-Dichloroethane	0.5	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Carbon Tetrachloride	0.5	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Benzene	0.5	0.408	6.530[a]	1.77	1.98	1.20	1.11
Trichloroethene	0.5	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Tetrachloroethene	0.7	< 0.002	< 0.004	< 0.040	< 0.040	< 0.040	< 0.040
Chlorobenzene	100	< 0.002	0.0101	< 0.040	< 0.040	< 0.040	< 0.040
1,4-Dichlorobenzene	7.5	0.0043 J	0.107	< 0.040	< 0.040	0.096 J	0.0904 J

[a] = Results from 2nd dilution of sample
J = Estimated value below the Method Reporting Limits (MRL)

Similar to the other leaching data collected, the SPLP testing of the thermally treated impoundment materials indicate that benzene is the only compound leached from the materials above the TCLP regulatory level. Benzene in the leachate from the thermally treated Impoundment 1 materials without pozzolan treatment was below the TCLP regulatory level. The concentration of benzene leached from the thermally treated and remixed Impoundment 1 materials was slightly greater than the benzene leached from the thermally treated and remixed Impoundment 2 materials. The leachate benzene concentration for the thermally treated and remixed Impoundment 2 materials was reduced up to 83.0% when comparted to the pre-solidified/stabilized thermally treated impoundment 2 materials. The benzene leaching from the solidified/stabilized thermally treated and remixed Impoundment 1 materials was greater than the benzene leached from the thermally treated Impoundment 1 materials. This anomaly may have resulted from incomplete homogenization of the thermally treated Impoundment 1 materials as described above.

Samples of the headspace air above the solidified/stabilized remixed thermally treated Impoundment materials was collected during the remixing step and after 28 days of curing. The results of the headspace sampling are located in Table 5-22.

TABLE 5-22

Laboratory Treatability Studies Report

Tier III Thermally Treated Impoundment Material Selected CoCs in Headspace During Remixing and 28-Day Cure

	IMP 1 TT Mix A Concentration (µg/m³)		IMP 1 TT Mix B Concentration (µg/m³)		IMP 2 TT Mix A Concentration (µg/m³)		IMP 2 TT Mix B Concentration (µg/m³)	
Compound	**Remixing**	**Day 28**	**Remixing**	**Day 28**	**Remixing**	**Day 28**	**Remixing**	**Day 28**
VOCs								
Benzene	1,100,000	690,000	1,200,000	240,000	770,000	650,000	310,000	840,000
Toluene	210,000	94,000	250,000	28,000	250,000	190,000	190,000	310,000
Naphthalene	< 6,800	< 3,300	< 7,000	< 1,200	< 4,500	< 4,000	< 2,300	< 5,000
Xylene (total)[1]	30,000	9,500	38,000	< 2,400	56,800	46,900	57,000	87,000
Ethylbenzene	< 6,800	< 3,300	< 7,000	< 1,200	4,600	< 4,000	4,400	7,100
Cumene	< 6,800	< 3,300	< 7,000	< 1,200	7,400	7,000	8,200	13,000
1,3,5-Trimethylbenzene	< 6,800	< 3,300	< 7,000	< 1,200	< 4,500	< 4,000	3,500	< 5,000
1,2,4-Trimethylbenzene	< 6,800	< 3,300	< 7,000	< 1,200	< 4,500	< 4,000	4,000	< 5,000
1,2-Dichlorobenzene	< 6,800	< 3,300	< 7,000	< 1,200	6,600	5,800	7,700	7,400
Aldehydes								
Acetaldehyde	10	100	20	30	40	30	40	140
Benzaldehyde	< 9	30	< 9	10	< 9	10	< 9	70
Butyraldehyde	< 10	10	< 10	< 8	< 10	< 8	< 10	40[2]
Crotonaldehyde	< 10	< 8	< 10	< 8	< 10	< 8	< 10	< 8
Formaldehyde	< 10	< 7	< 10	< 7	< 10	10	10	10
Isovaleraldehyde	< 10	< 7	70[2]	< 7	90[2]	< 7	120[2]	< 7
Propionaldehyde	< 10	< 7	< 10	< 7	< 10	< 7	< 10	20
Valeradehyde	< 10	< 7	< 10	< 7	< 10	< 7	< 10	< 7
Acid Gases								
Sulfuric Acid	< 1,000	< 80	< 1,000	< 80	< 1,000	< 80	< 1,000	< 80
Malodorous Compounds								
Hydrogen Sulfide	< 7	< 7	< 7	< 7	< 7	< 7	< 7	< 7
Carbonyl Sulfide	33	23	< 12	17	41	59	51	31
Methyl Mercaptan	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Dimethyl Sulfide	200	210	38	170	460	640	680	280
Carbon disulfide	4,400	2,300	1,300	1,800	4,100	4,400	4,000	4,100
Ethyl Methyl Sulfide	35	29	< 16	20	89	89	110	46
Thiophene	220	310	70	190	1,900	1,100	2,200	1,400
3-Methylthiophene	45	58	< 20	37	380	300	430	320
2-Ethylthiophene	34	50	< 23	26	740	560	840	620

Notes:

[1.] Total xylene calculated by the sum of *ortho, meta, and para* –xylene concentration detected.

[2.] Sorbent tube may have had breakthrough or possible migration. Results may be biased low.

The concentrations of compounds detected in the headspace air above the remixed and 28-day cured remixed thermally treated impoundment materials was lower than the compounds detected in the remixed and 28-day cured remixed impoundment materials that were not thermally treated. During the remixing of the thermally treated impoundment materials, the headspace air above the Impoundment 1 materials contained higher concentration than the headspace air above the Impoundment 2 materials. The extent of reduction in VOC concentration from the mixing step to the day 28 cure period was less for the solidified/stabilized and remixed thermally treated impoundment materials than for the solidified/stabilized and remixed impoundment materials that were not thermally treated. The remixed thermally treated Impoundment 2 materials that were initially solidified/stabilize using Mix B showed an increase in headspace VOCs, some aldehydes and some malodorous compounds from the remix to the day 28 cure period.

5.5 Tier IV Treatability Study

Tier IV treatability studies were conducted to optimize the pozzolan recipes to evaluate the potential for additional permeability reduction, COC leaching reduction and COC volatilization reduction. Evaluation of the patented Dispersion by Chemical Reaction (DCR) technology was proposed to be conducted during the additional Tier IV screening process per the recommendation of the United States Environmental Protection Agency (USEPA) as an additional option for stabilization of impoundment material. However, the patent holder, Tasmania Limited, was unresponsive to requests to obtain a license to evaluate the technology. The additional treatability studies were conducted on both raw and thermally treated materials from Impoundment 2. The results of the Tier IV treatability studies are attached as Appendix G.

SECTION 6

Conclusions

6.1 Thermal Treatment

The treatability study confirmed that controlled heating of impoundment materials collected from Impoundment 2 at the Site was successful in significantly reducing VOCs and SVOCs content of VR and HC materials.

This portion of the treatability study performed was a comprehensive evaluation intended to investigate the effectiveness of thermal processes for the treatment of materials contained within Impoundment 1 and Impoundment 2. The study was premised on two basic objectives: to determine the efficacy of thermal treatment for impoundment materials and to identify and characterize both off-gas and liquid-phase condensate that must be managed if thermal treatment was implemented for treating impoundment contents. The study objectives were successfully accomplished as documented in the extensive observations and data results provided by this report and supporting appendixes.

In general, this study reveals that heating was successful in significantly reducing VOCs and SVOCs in the impoundment materials studied. Highly acidic off-gas caused significant corrosion of the box reactor, aluminum lining, and thermocouples used during the thermal treatment. Treatment of the material for extended periods of time did not completely dry out the sample. Some physical properties testing were complicated and affected by the high concentrations of VOCs and the matrix of the material before and after heating. Even after thermal treatment, VR material that was reheated exhibited liquid behavior as temperatures reached approximately 65°C. The HC material does not show signs of liquefaction up to temperatures of 100°C. The VR material shows significant signs of expansion during heating, while the HC material shows moderate expansion.

6.2 Mixing, pH Adjustment, and Solidification/Stabilization

The treatability study confirmed that the materials from Impoundment 1 and 2 at the Site could be successfully homogenized, pH adjusted, and solidified using pozzolans.

The basic objectives of this portion of the treatability study were:

1) Evaluate impoundment materials for homogenization
2) Determine the pH of the impoundment materials could be raised to at least 5 to 6 SU and ideally to 11 SU.
3) Evaluate if the impoundment materials could be stabilized/solidified using pozzolan mixtures.

In general, the results of the study indicate that the impoundment materials can be successfully homogenized, pH adjusted and solidified. Results from this study confirmed that both raw Impoundment 1 and 2 materials could be homogenized; however, the water layer on the raw Impoundment 2 material did not become incorporated with the material. Homogenized samples of materials from the Impoundments 1 and 2 were successfully pH adjusted to 10 SU or higher by an addition of Carmeuse Hydrated Lime or HiCal LKD. Addition of various blends of pozzolans to the homogenized and pH adjusted impoundment material resulted in strength gains ranging from 59.0 psi to greater than 62.5 psi after only seven days of curing.

Tier II pozzolan testing of thermally treated Impoundment 1 and 2 materials indicated that the concentration of benzene in the atmosphere above the mixing vessel decreased by approximately two orders of magnitude when compared to benzene concentration above the raw impoundment materials during mixing. When the Tier II solidified/stabilized materials were crushed to simulate excavation from the impoundments, secondary pozzolan reagents and water added and the materials remolded to simulate placement in a landfill, significant strength was achieved. This step validated the need for an additional remixing step being required to increase the materials strength and maximize potential disposal options. The Tier III remixing resulted in significant UCS gains over the Tier II UCS results.

Tier III testing results indicated that additional studies should be conducted under a Tier IV evaluation program to attempt to lower the permeability and VOC emissions of the final treated impoundment materials. The Tier III blends that were incorporated into the Tier IV testing are located in Table 6-1.

TABLE 6-1
Laboratory Treatability Studies Report
Best Performing ISS Recipes Identified During Tier I through Tier III of the Treatability Studies

Material Type	Mixture	pH Buffering	De-emulsification	Initial Solidification	Remix / Stabilization
Raw	IMP 2 RM MIX A	6% Hydrated Lime/ 15% water	-	33% SFE / 15% LN / 15% Omni FBC	10% LPC / 8% water
	IMP 2 RM MIX B	6% Hydrated Lime/ 15% water	-	33% SFE / 30% LN	10% LPC / 8% water
Thermally Treated	IMP 2 TT MIX A	-	30% HiCal LKD / 30% water	15% H_2O / 5% LPC / 5% Omni FBC	10% LPC / 8% water
	IMP 2 TT MIX B	-	30% HiCal LKD / 30% water	15% H_2O / 10% LN	10% LPC / 8% water

Notes:
FBC = fluidized bed combustion ash
LN = LaFarge NewCem
LPC = LaFarge portland cement
SFE = spent fullers earth (screened)

The Tier IV Studies were completed and a separate report was prepared and is located in Appendix G.

SECTION 7

References

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